U.S. DEPARTMENT OF COMMERCE
National Technical Information Service
PB-274 320

Effluent Monitoring Procedures Nutrients. Staff Guide for Conducting the Course

National Training & Operational Technology Ctr, Cincinnati, Ohio

Nov 77

PB 274 320

EPA-430/1-77-007

EFFLUENT MONITORING PROCEDURES: NUTRIENTS

STAFF GUIDE

U.S. ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF WATER PROGRAM OPERATIONS

REPRODUCED BY
NATIONAL TECHNICAL
INFORMATION SERVICE
U. S. DEPARTMENT OF COMMERCE
SPRINGFIELD, VA. 22161

BIBLIOGRAPHIC DATA SHEET

1. Report No. EPA-430/1-77-007

2.

3. Recipient's Accession No. PB274320

4. Title and Subtitle

Effluent Monitoring Procedures: Nutrients
Staff Guide

5. Report Date November 1977

6.

7. Author(s) Audrey D. Kroner

8. Performing Organization Rept. No.

9. Performing Organization Name and Address

Environmental Protection Agency
Municipal Operations & Training Division
National Training & Operational Technology Center
Cincinnati, Ohio 45268

10. Project/Task/Work Unit No.

11. Contract/Grant No.

12. Sponsoring Organization Name and Address

Same as No. 9 above

13. Type of Report & Period Covered Final

14. 700/02

15. Supplementary Notes

16. Abstracts

This manual is designed for use by administrators, support staff and instructors who will be responsible for presenting the topics in the student reference text, "Effluent Monitoring Procedures: Nutrients" EPA-430-1-76-006. It contains information about course planning and management: course plan and working schedules; required staff, facilities, equipment, supplies, and instructional resources; course registration, record-keeping, and attendant printed materials. It also contains instructional package worksheets (lesson plans) for each topic in the student reference text.

17. Key Words and Document Analysis. 17a. Descriptors

Analysis, analytical techniq ues, chemical analysis, education, training, ammonia, graphs, greases, inorganic nitrates- nitrites, nitrogen, oils, oxygen demand, phosphorus, spectrophotometers.

17b. Identifiers/Open-Ended Terms

Effluent Monitoring Procedures

17c. COSATI Field/Group 14B, 07B

18. Availability Statement

Release to the Public

19. Security Class (This Report) UNCLASSIFIED

20. Security Class (This Page) UNCLASSIFIED

21. No. of Pages 213

22. Price PC A10-A01

FORM NTIS-35 (REV. 3-72)

USCOMM-DC 14952-P72

STAFF GUIDE
for
CONDUCTING THE COURSE

EFFLUENT MONITORING PROCEDURES: NUTRIENTS

National Training and Operational Technology Center
Municipal Operations and Training Division
Office of Water Program Operations
U.S. Environmental Protection Agency

ia

DISCLAIMER

Reference to commercial products, trade names, or manufacturers is for purposes of example and illustration. Such references do not constitute endorsement by the Office of Water Program Operations, U.S. Environmental Protection Agency.

COPYRIGHT INFORMATION

The contents of this manual may be copied freely, in whole or in part, without violation of copyright laws provided that: when copies are made, reference to the U. S. Government Printing Office, on the last page of this manual, is deleted.

TABLE OF CONTENTS

Page

PREFACE TO THE USER OF THIS MANUAL 1

Part I COURSE PLANNING AND MANAGEMENT

A. COURSE PLAN AND WORKING SCHEDULES 1-1
  Responsibilities in Self-Monitoring Training 1-1
  Announcing the Course 2-1
  Summary Plan for the Course 3-1
  Sample Course Schedule 4-1
  Milestones in Course Planning and Preparation 5-1

B. TRAINING RESOURCES 6-1
  Training Staff 6-1
  Training Facilities 7-1
  Laboratory Equipment and Supply Requirements 8-1
  Instructional Resources 9-1

C. SECRETARIAL SUPPORT 10-1
  Course Records and Record-keeping 10-1
  Suggested Student Registration Procedures 11-1
  Printed and Reproduced Materials-Summary 12-1

Part II INSTRUCTIONAL PACKAGE WORKSHEETS 13-1

A. Use of a Spectrophotometer 14-1

B. Preparation of Calibration Graphs 15-1

C. Determination of Total Phosphorus (as P) or of Orthophosphate (as P), Single Reagent Method 16-1

D. Determination of Chemical Oxygen Demand 17-1

E. Determination of Total Kjeldahl Nitrogen 18-1

F. Nitrogen, Ammonia Determination 19-1

G. Determination of Nitrate-Nitrite Nitrogen and of Nitrate Nitrogen, Cadmium Reduction Method 20-1

H. Determination of Oil and Grease 21-1

I. Determination of Ammonia by an Ammonia Selective Ion Electrode 22-1

PREFACE

TO THE USER OF THIS MANUAL

Background of Effluent Self-Monitoring Requirement

With passage of the Federal Water Pollution Control Act Amendments of 1972 (Public Law 92-500) a new permit program was created to replace and improve upon the earlier permit system which existed under the 1899 Refuse Act.

Under the 1972 Act, the United States Environmental Protection Agency is required to establish national effluent limitations and national treatment performance standards for all sources of water pollution, including not only municipal discharges, but also factories, animal feedlots, and power plants. These effluent limitations are the maximum amount of a pollutant that any discharger may release into a water body.

In order to insure that the prescribed effluent limits are met, every discharger is required to obtain an NPDES (National Pollutant Discharge Elimination System) Permit. Types of water discharge sources for which a permit is required include municipal wastewater treatment facilities; manufacturing plants; agriculture, forestry, mining and fishing operations, and other service, wholesale, retail, and commercial establishments having operations which result in discharge of water to the Nation's bodies of water.

The NPDES Permit is not a license to pollute. To the contrary, a Permit stipulates what may be discharged, and how much may be discharged over a defined period of time. Each Permit is tailored to the discharger, and sets specific limits on each effluent.

Furthermore, the NPDES Permit also requires dischargers to monitor their effluents, performing specified tests and measurements at designated frequencies specified in the individual Permit, and to report the amount and nature of all waste components discharged.

Training Need Associated with Self-Monitoring Requirements

Compliance with NPDES requires that all specified tests and measurements be performed in accordance with methods specified by EPA and announced in the Federal Register. Only under very limited conditions are deviations from specified methods acceptable.

These requirements are the basis of an immediate, massive, training need to provide the responsible waste water treatment personnel with the knowledge and skills required to comply with the self-monitoring requirement. There is at present a wide range of initial capabilities for performing the tests and measurements. This ranges from the situation in the large, fully-staffed, fully-equipped facility in which little or no training is required, through all shades and levels to the limited staff in small, inadequately-equipped facilities in which at present there is little or no capability for performing the required tests and measurements.

This is one of several special short-term courses recently developed and currently under development by the Environmental Protection Agency and associated educational institutions. This Instructor's Guide is intended to assist other training organizations in conducting training of waste water treatment plant operational personnel in the tests, measurements, and report preparation required for compliance with their NPDES Permits.

TRAINING AVAILABLE OR UNDER DEVELOPMENT TO MEET SELF-MONITORING REQUIREMENTS

Title	Workers should take this who:
Basic Laboratory Skills for Self-Monitoring Tests and Measurements	Have little or no laboratory experience or training, and need to develop basic skills including use of balances; preparation of solutions and reagents; names, characteristics, preparation and care of common laboratory equipment supplies. This course prepares students requiring such training for entry into the following named courses in which procedures for designated tests and measurements are learned.
Self-Monitoring Procedures: Basic Parameters for Municipal Effluents	Have basic laboratory skills, as designated above, but need to learn one or more of the procedures required for most or all municipal effluents including BOD, fecal coliform, pH, suspended solids, flow, and reporting of results.
Effluent Monitoring Procedures: Metals Analyses	Usually have completed the "Basic Parameters" course, but have a Permit which requires report on one or more metals.
Effluent Monitoring Procedures: Nutrients	Usually have completed the "Basic Parameters" course, but have a Permit which requires periodic report on one or more of the so-called "nutrients" including nitrogen and phosphorus.
Effluent Monitoring Procedures: Flow Measurement and Sampling Techniques	Have made arrangements whereby the required laboratory procedures are performed elsewhere by contract or other special arrangement, and who are required only to collect and care for samples, and to make such tests and measurements as must be made at the sampling site.
Any of the analytical courses of EPA National Training and Operational Technology Center for specific Permits	Are professional chemists, microbiologists, or key laboratory technicians in large treatment facilities where they work full-time doing a designated series of analytical tests and measurements.

A Personal Note to the User of this Guide

The need for providing this training, and the nature of current efforts of the Environmental Protection Agency to meet this need have been discussed in foregoing paragraphs.

It is an oversimplification of a classic quotation to say that all that is needed to conduct training is to "put Mark Hopkins on one end of a log and a student on the other." It is almost as much an oversimplification to say that all that is needed to conduct a course is to bring together the students and the instructional staff in a classroom and laboratory equipped with requisite equipment and supplies for the instruction to be delivered.

A short course such as this, involving both classroom instruction and laboratory application, and with critical learning outcomes to be achieved within a limited period of time, absolutely requires careful, detailed planning, preparation, and implementation. Meticulous attention to detail and effective staff teamwork are essential at every step of course planning, development, and implementation.

The purpose of this Staff Guide is to provide you, the training staff member, with useful, practical assistance in presenting this course in your own facilities. The Guide is a resource. It is not a blueprint to be followed rigidly or unthinkingly. Even with this guide or any other form of assistance, you will have to plan, to think and to prepare in order to perform effectively in conducting this course. On the other hand, this Guide should be helpful in reducing the amount of original development work you will have to do, and it should be helpful in suggesting factors in course planning and presentation which otherwise might be ignored or glossed over.

You are invited, in fact requested, to participate actively in making this Guide a living document which effectively represents the best experience of all in planning, preparing for, conducting and terminating this course. Please provide this office with your constructive suggestions for strengthening and improving upon this Guide, based on your own experiences in conducting the course. Your recommendations will be fully considered in future editions of this and other Staff Guides for other courses of this series. In the event that questions arise in interpretation of any aspect of this Instructors' Guide, please write or call:

Director, National Training and Operational Technology Center
Office of Water Program Operations
U.S. Environmental Protection Agency
Cincinnati, OH 45268

Telephone: (513) 684-7501

Format of this Manual

This manual consists of two major parts. Part I is concerned with administrative aspects of planning, preparing, and conducting the course. Part II consists of guidance to the instructional staff on the technical content, learning objectives to be achieved by the students, and lesson-by-lesson instructional guides for each of the several self-monitoring procedures covered in this course.

Part I, which immediately follows, will have greatest value to those conducting this course for the first time or for the first few times. With practice in conducting the training most organizations will develop their own adaptations and ramifications of this plan to meet their own requirements and the specific situation.

It is strongly urged that each institution conducting this training course develop its own formal version of such a guide as this. Staff does change; new personnel require indoctrination and training on standard procedures of the organization they have joined. Rules and procedures promulgated by regulatory authority do change; such changes will require adjustment in course content. With repetition of specified tasks, many staff members, however dedicated, do slip into unconscious variations in practice or omissions in details which can subtly change the character of the course. A formal plan and guide, consciously studied and followed, can help the training instructor and the administrator to avoid many pitfalls.

Intelligent use of a plan such as this, with variations as proven necessary, can go far in keeping this course in harmony with the needs of the students and with requirements of regulatory authorities. Above all, the course should remain free of the distractions and last-minute corrections of errors or omissions discovered at untimely stages in course preparation and presentation.

PART I - COURSE PLANNING AND MANAGEMENT

A. Course Plan and Working Schedules

This section considers five topics:

Responsibilities in self-monitoring training

It is an inherent responsibility of any teacher to provide a learning situation which gives the student the best possible opportunity to develop the target level of knowledge and skills.

In addition, and not to be overlooked, this course involves a responsibility to the regulatory agency--to assure that students acquire specified knowledge and skills and that these skills are acquired to a level such that data reported by the student will be accepted as reliable by the regulatory authority.

Announcing the course

This section gives examples of course announcements, and identifies specific information which should be provided in any course announcement.

Summary plan for the course

This is a one-page summary of the course plan in which the reader can discover the subject matter coverage, days and approximate time allocations and the designation of the instructional specialty involved in presenting the instruction.

Sample course schedule

This covers the same information as the summary plan. The format is different, providing a day-by-day, hour-by-hour class schedule. This schedule-format has been found most practical at EPA training centers for more than 20 years. It works. It is recommended for your use.

Milestones in course planning and preparation

Each member of the training staff has individual and cooperative duties in planning and conducting the course. Much of the responsibility of each staff member is outlined later in this Guide in the section on Training Staff (Outline 6). The milestone chart shown here is an example of a plan to accomplish necessary tasks in a timely manner. It is necessary that each training institution develop its own logistics of course preparation to meet the situation at hand. It is urged that a formal milestone chart be developed.

1. Responsibilities in Self-Monitoring Training

a. Implications of NPDES

Each Permit issued under the National Pollutant Discharge Elimination System (NPDES) includes a program of required self-monitoring analyses of effluents and reporting of results at prescribed intervals.

1) The methodology to be followed in performing the self-monitoring tests and measurements is described in issuances in the Federal Register (FR).

2) In some cases, two or more alternative procedures are available to the analyst for compliance with monitoring requirements.

3) Provisions do exist whereby the regulatory agencies (State and EPA) can recommend and permit use of methods not listed in the FR. Procedures for orderly application of accepted methods are described in the FR issuance at the end of this section. It cannot be too strongly emphasized that the Permit-holder does not have the authority to make a unilateral decision to introduce analytical procedures not found in the FR issuances or not specifically authorized by the appropriate regulatory authorities.

b. Training Response to NPDES/FR Requirements

1) Methods taught in this and associated courses developed by EPA are limited to those most recently prescribed in issuances of the FR.

2) Usually, when alternative methods are available, the course will include only one of the alternatives. In planning the details of a specific course offering, a training representative should in every case consult with responsible representatives of the regulatory agency in the area in which the course is conducted. This consultation should include determination of:

   a) Which, if any, of more than one alternative analytical method is preferred (or required) by the regulatory agency for the Permit-holders under its jurisdiction?

   b) What, if any, changes have occurred since the last course offerings which require adjustment in course content?

   c) What advice does the regulatory agency have to offer about the planned course presentation based on problems encountered in implementation of self-monitoring procedures, and in observations of performance of past graduates from previous offerings of the course?

3) The training institution which fails to apply these elementary practices, with a result of sometimes teaching inappropriate methods, stands in danger of committing a serious disservice to those it purports to serve:

   a) To the students enrolled in the course; and

   b) To the Permit-holding organization, which is being led to believe that through this training the qualifying student will perform self monitoring tests and measurements in accordance with methods approved by the regulatory authority.

c. Responsibilities in Accreditation of Students

1) Successful completion of this course of training will be a factor used by many regulatory agencies in the accreditation or certification of treatment plant personnel to perform self-monitoring tests and measurements.

2) The instructional staff has a responsibility to provide a learning experience through which the qualifying student will have every expectation of being able to perform the required self-monitoring procedures in a satisfactory manner on return to his normal working environment.

3) Student Evaluation and Testing

For permanent record of qualification and performance, a record of student performance should be made.

a) Tests (both written and applied) should be criterion tests, i.e., designed to demonstrate ability of the student to perform the required analyses, as contrasted with tests designed to develop a comparative rating of the individual students.

b) Written tests should be strictly applied to the procedures being taught and should be appropriate to a written answer. Thus definitions, matters of specific information, solving of problems, and similar matters are appropriate to written tests.

c) Many students will take alarm at written tests, and may not perform up to expectations due to this alarm. One means of alleviating this fear is to give open-book quizzes but to expect a high standard of performance. It is pointed out here that it is more important for the student to know where to refer for needed facts and to check these facts, than it is to demonstrate rote memory. The memory will come with practice of the test procedures. The student should learn to check the facts when in doubt, rather than to risk a blunder.

d) Much of the student evaluation will be based on performance in the laboratory. It will be appropriate to keep records on such factors in student performance as:

(1) Accuracy in following directions as given;

(2) Demonstration of acceptable manipulative skills;

(3) Reporting of laboratory results falling within acceptable relationship to a class norm; and other factors as determined by the instructor.

e) Each instructor is responsible for reporting the quality of performance of each student for the procedures for which he has primary responsibility. These reports should be written, and they should be made a part of the permanent course record. This course plan provides for having at least two instructors in the laboratory during all laboratory phases of the course. At any given session, one instructor will be the designated primary instructor, with others in a supporting role. To make a system like this work satisfactorily in evaluation of students and to assure consistence in student instruction each member of the instructional team has defninte, if implied, responsibilities. These include:

(1) The primary instructor is the individual who reports on acceptability or non-acceptability of student performance.

(2) The instructor-assistant(s) must instruct students in the same way as designated by the primary instructor. Introduction of individuality in manipulative procedures, order of proceeding with a test, etc., can only confuse the student.

(3) The instructor-assistant(s) should bring the problem-student to the attention of the primary instructor at the earliest possible moment. This is to provide maximum opportunity to correct problems which might result in failure of the student to qualify in a given analytical procedure.

(4) Primary instructor and instructor-assistants have, of course, a continuing responsibility of preserving the highest standards of professional and ethical relationships with each other throughout the course. Differences of opinion will arise among individuals of any instructional staff. These differences must be resolved outside the classroom and laboratory. Furthermore, these differences are not subjects for airing with students at any time.

4) The Nonqualifying Student

a) There is a job/financial implication to any student failing to qualify for any of the analytical tests and measurements included in this course. Therefore instructors should be particularly careful to document the reasons for any nonqualification.

b) Any nonqualifying student should be entitled to be informed on the reasons why he is judged nonqualifying.

c) Ideally, an opportunity should be provided for nonqualifying students to have another chance to correct deficiencies in their performance. This could be accomplished through a number of different approaches such as makeup work (evenings) during the course itself, through repeating the training module in which acceptable results were not achieved (at a mutually agreeable time for the student and for the instructor), or by enrollment in a future offering of the course with participation only in the module which was failed the first time around.

federal register

WEDNESDAY, DECEMBER 1, 1976

THE NATIONAL ARCHIVES OF THE UNITED STATES • 1934

PART II:

ENVIRONMENTAL PROTECTION AGENCY

■

WATER PROGRAMS

Guidelines Establishing Test Procedures for the Analysis of Pollutants

Amendments

Title 40—Protection of Environment

CHAPTER I—ENVIRONMENTAL PROTECTION AGENCY

SUBCHAPTER D—WATER PROGRAMS

[FRL 630-4]

PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

Amendment of Regulations

On June 9, 1975, proposed amendments to the Guidelines Establishing Test Procedures for the Analysis of Pollutants (40 CFR 136) were published in the Federal Register (40 FR 24535) as required by section 304(g) of the Federal Water Pollution Control Act Amendments of 1972 (86 Stat. 816, et seq., Pub. L. 92–500, 1972) hereinafter referred to as the Act.

Section 304(g) of the Act requires that the Administrator shall promulgate guidelines establishing test procedures for the analysis of pollutants that shall include factors which must be provided in: (1) any certification pursuant to section 401 of the Act, or (2) any permit application pursuant to section 402 of the Act. Such test procedures are to be used by permit applicants to demonstrate that effluent discharges meet applicable pollutant discharge limitations and by the States and other enforcement activities in routine or random monitoring of effluents to verify compliance with pollution control measures.

Interested persons were requested to submit written comments, suggestions, or objections to the proposed amendments by September 7, 1975. One hundred and thirty-five letters were received from commenters. The following categories of organizations were represented by the commenters: Federal agencies accounted for twenty-four responses; State agencies accounted for twenty-six responses; local agencies accounted for seventeen responses; regulated major dischargers accounted for forty-seven responses; trade and professional organizations accounted for eight responses; analytical instrument manufacturers and vendors accounted for seven responses; and analytical service laboratories accounted for six responses.

All comments were carefully evaluated by a technical review committee. Based upon the review of comments, the following principal changes to the proposed amendments were made:

(A) *Definitions.* Section 136.2 has been amended to update references: Twenty commenters, representing the entire spectrum of responding groups pointed out that the references cited in §§ 136.2(f), 136.2(g), and 136.2(h) were out-of-date; §§ 136.2(f), 136.2(g), and 136.2(h), respectively, have been amended to show the following editions of the standard references: "14th Edition of Standard Methods for the Examination of Water and Waste Water;" "1974 EPA Manual of Methods for the Analysis of Water and Waste;" and "Part 31, 1975 Annual Book of ASTM Standards."

(B) *Identification of Test Procedures.* Both the content and format of § 136.3, "Table I, List of Approved Test Procedures" have been revised in response to twenty-one comments received from State and local governments, major regulated dischargers, professional and trade associations, and analytical laboratories.

Table I has been revised by:

(1) The addition of a fourth column of references which includes procedures of the United States Geological Survey which are equivalent to previously approved methods.

(2) The addition of a fifth column of miscellaneous references to procedures which are equivalent to previously approved methods.

(3) Listing generically related parameters alphabetically within four subcategories: bacteria, metals, radiological and residue, and by listing these subcategory headings in alphabetic sequence relative to the remaining parameters.

(4) Deleting the parameter "Algicides" and by entering the single relevant algicide, "Pentachlorophenol" by its chemical name.

(C) *Clarification of Test Parameters.* The conditions for analysis of several parameters have been more specifically defined as a result of comments received by the Agency:

(1) In response to five commenters representing State or local governments, major dischargers, or analytical instrument manufacturers, the end-point for the alkalinity determination is specifically designated as pH 4.5.

(2) Manual digestion and distillation are still required as necessary preliminary steps for the Kjeldahl nitrogen procedure. Analysis after such distillation may be by Nessler color comparison, titration, electrode, or automated phenolate procedures.

(3) In response to eight commenters representative of Federal and State governments, major dischargers, and analytical instrument manufacturers, manual distillation at pH 9.5 is now specified for ammonia measurement.

(D) *New Parameters and Analytical Procedures.* Forty-four new parameters have been added to Table I. In addition to the designation of analytical procedures for these new parameters, the following modifications have been made in analytical procedures designated in response to comments.

(1) The ortho-tolidine procedure was not approved for the measurement of residual chlorine because of its poor accuracy and precision. Its approval had been requested by seven commenters representing major dischargers, State, or local governments, and analytical instrument manufacturers. Instead, the N,N-diethyl-p-phenylenediamine (DPD) method is approved as an interim procedure pending more intensive laboratory testing. It has many of the advantages of the ortho-tolidine procedure such as low cost, ease of operation, and also is of acceptable precision and accuracy.

(2) The Environmental Protection Agency concurred with the American Dye Manufacturers' request to approve its procedure for measurement of color, and copies of the procedure are now available at the Environmental Monitoring and Support Laboratory, Cincinnati (EMSL–CI)

(3) In response to three requests from Federal, State governments, and dischargers, "hardness," may be measured as the sum of calcium and magnesium analysed by atomic absorption and expressed as their carbonates.

(4) The proposal to limit measurement of fecal coliform bacteria in the presence of chlorine to only the "Most Probable Number" (MPN) procedure has been withdrawn in response to requests from forty-five commenters including State pollution control agencies, permit holders, analysts, treatment plant operators, and a manufacturer of analytical supplies. The membrane filter (MF) procedure will continue to be an approved technique for the routine measurement of fecal coliform in the presence of chlorine. However, the MPN procedure must be used to resolve controversial situations. The technique selected by the analyst must be reported with the data.

(5) A total of fifteen objections, representing the entire spectrum of commenters, addressed the drying temperatures used for measurement of residues. The use of different temperatures in drying of total residue, dissolved residue and suspended residue was cited as not allowing direct intercomparability between these measurements. Because the intent of designating the three separate residue parameters is to measure separate waste characteristics (low drying temperatures to measure volatile substances, high drying temperatures to measure anhydrous inorganic substances), the difference in drying temperatures for these residue parameters must be preserved.

(E) *Deletion of Measurement Techniques.* Some measurement techniques that had been proposed have been deleted in response to objections raised during the public comment period.

(1) The proposed infrared spectrophotometric analysis for oil and grease has been withdrawn. Eleven commenters representing Federal or State agencies and major dischargers claimed that this parameter is defined by the measurement procedure. Any alteration in the procedure would change the definition of the parameter. The Environmental Protection Agency agreed.

(2) The proposed separate parameter for sulfide at concentrations below 1 mg/l, has been withdrawn. Methylene blue spectrophotometry is now included in Table I as an approved procedure for sulfide analysis. The titrimetric iodine procedure for sulfide analysis may only be used for analysis of sulfide at concentrations in excess of one milligram per liter.

(F) *Sample Preservation and Holding Times.* Criteria for sample preservation and sample holding times were requested by several commenters. The reference for sample preservation and holding time criteria applicable to the Table I parameters is given in footnote (1) of Table I.

(G) *Alternate Test Procedures.* Comments pertaining to § 136.4, Application for Alternate Test Procedures, included objections to various obstacles within these procedures for expeditious approval of alternate test procedures. Four analytical instrument manufacturers commented that by limiting of application for review and/or approval of alternate test procedures to NPDES permit holders, § 136.4 became an impediment to the commercial development of new or improved measurement devices based on new measurement principles. Applications for such review and/or approval will now be accepted from any person. The intent of the alternate test procedure is to allow the use of measurement systems which are known to be equivalent to the approved test procedures in waste water discharges.

Applications for approval of alternate test procedures applicable to specific discharges will continue to be made only by NPDES permit holders, and approval of such applications will be made on a case-by-case basis by the Regional Administrator in whose Region the discharge is made.

Applications for approval of alternate test procedures which are intended for nationwide use can now be submitted by any person directly to the Director of the Environmental Monitoring and Support Laboratory in Cincinnati. Such applications should include a complete methods write-up, any literature references, comparability data between the proposed alternate test procedure and those already approved by the Administrator. The application should include precision and accuracy data of the proposed alternate test procedure and data confirming the general applicability of the test procedure to the industrial categories of waste water for which it is intended. The Director of the Environmental Monitoring and Support Laboratory, after review of submitted information, will recommend approval or rejection of the application to the Administrator, or he will return the application to the applicant for more information. Approval or rejection of applications for test procedures intended for nationwide use will be made by the Administrator, after considering the recommendation made by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati. Since the Agency considers these procedures for approval of alternate test procedures for nationwide use to be interim procedures, we will welcome suggestions for criteria for approval of alternate test procedures for nationwide use. Interested persons should submit their written comments in triplicate on or before June 1, 1977 to: Dr. Robert B. Medz, Environmental Protection Technologist, Monitoring Quality Assurance Standardization, Office of Monitoring and Technical Support (RD-680), Environmental Protection Agency, Washington, D.C. 20460.

(H) *Freedom of Information.* A copy of all public comments, an analysis by parameter of those comments, and documents providing further information on the rationale for the changes made in the final regulation are available for inspection and copying at the Environmental Protection Agency Public Information Reference Unit, Room 2922, Waterside Mall, 401 M Street, SW., Washington, D.C. 20460, during normal business hours. The EPA information regulation 40 CFR 2 provides that a reasonable fee may be charged for copying such documents.

Effective date: These amendments become effective on April 1, 1977.

Dated: November 19, 1976.

JOHN QUARLES,
Acting Administrator,
Environmental Protection Agency.

Chapter I, Subchapter D, of Title 40, Code of Federal Regulations is amended as follows:

1. In § 136.2, paragraphs (f), (g), and (h) are amended to read as follows:

§ 136.2 Definitions.

* * * * *

(f) "Standard Methods" means *Standard Methods for the Examination of Water and Waste Water*, 14th Edition, 1976. This publication is available from the American Public Health Association, 1015 18th Street, N.W., Washington, D.C. 20036.

(g) "ASTM" means *Annual Book of Standards, Part 31, Water, 1975.* This publication is available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

(h) "EPA Methods" means *Methods for Chemical Analysis of Water and Waste, 1974.* Methods Development and Quality Assurance Research Laboratory, National Environmental Research Center, Cincinnati, Ohio 45268; U.S. Environmental Protection Agency, Office of Technology Transfer, Industrial Environmental Research Laboratory, Cincinnati, Ohio 45268. This publication is available from the Office of Technology Transfer.

2. In § 136.3, the second sentence of paragraph (b) is amended, and a new paragraph (c) is added to read as follows:

§ 136.3 Identification of test procedures.

* * * * *

(b) * * * Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator or the Director upon the recommendation of the Director of the Environmental Monitoring and Support Laboratory, Cincinnati.

(c) Under certain circumstances, the Administrator may approve, upon recommendation by the Director, Environmental Monitoring and Support Laboratory, Cincinnati, additional alternate test procedures for nationwide use.

3. Table I of § 136.3 is revised by listing the parameters alphabetically; by adding 44 new parameters; by adding a fourth column under references listing equivalent United States Geological Survey methods; by adding a fifth column under references listing miscellaneous equivalent methods; by deleting footnotes 1 through 7 and adding 24 new footnotes, to read as follows:

* * * * *

TABLE I.—*List of approved test procedures*[1]

Parameter and units	Method	References (page nos.) 1974 EPA methods	14th ed. standard methods	Pt. 31 1975 ASTM	USGS methods[2]	Other approved methods
1. Acidity, as $CaCO^3$, milligrams per liter.	Electrometric end point (pH of 8.2) or phenolphthalein end point.	1	273(4d)	116	40	[3](607)
2. Alkalinity, as $CaCO^3$, milligrams per liter.	Electrometric titration (only to pH 4.5) manual or automated, or equivalent automated methods.	3	278	111	41	[3](607)
		5				
3. Ammonia (as N), milligrams per liter.	Manual distillation[4] (at pH 9.5) followed by nesslerization, titration, electrode, Automated phenolate.		410			
		159	412	237	116	[3](614)
		165				
		168	616			
BACTERIA						
4. Coliform (fecal)[4], number per 100 ml.	MPN;[5] membrane filter		922			
			937		[7](45)	
5. Coliform (fecal)[4] in presence of chlorine, number per 100 ml.	do.[5][6]		922			
			928, 937			
6. Coliform (total),[4] number per 100 ml.	do.[5]		916			
			928		[7](35)	
7. Coliform (total)[4] in presence of chlorine, number per 100 ml.	MPN;[5] membrane filter with enrichment.		916			
			933			
8. Fecal streptococci,[4] number per 100 ml.	MPN;[5] membrane filter; plate count.		943			
			944		[7](50)	
			947			
9. Benzidine, milligrams per liter.	Oxidation—colorimetric[8]					
10. Biochemical oxygen demand, 5-d (BOD_5), milligrams per liter.	Winkler (Azide modification) or electrode method.		543		[7](50)	
						[10](17)
11. Bromide, milligrams per liter.	Titrimetric, iodine-iodate	14		323	58	
12. Chemical oxygen demand (COD), milligrams per liter.	Dichromate reflux	20	550	472	124	[3](610)
						[10](17)
13. Chloride, milligrams per liter.	Silver nitrate; mercuric nitrate; or automated colorimetric-ferricyanide.		303	267		
		29	304	265		[3](615)
		31	613		[11](46)	

See footnotes at end of table.

Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.)		Other approved methods
				Pt. 31 1975 ASTM	USGS methods [5]	
14. Chlorinated organic compounds (except pesticides), milligrams per liter.	Gas chromatography [12].					
15. Chlorine—total residual, milligrams per liter.	Iodometric titration, amperometric or starch-iodine end-point; DPD colorimetric or Titrimetric methods (these last 2 are interim methods pending laboratory testing).	 35	318 322 332 329	 278		
16. Color, platinum cobalt units or dominant wave length, hue, luminance, purity.	Colorimetric; spectrophotometric; or ADMI procedure.[13]	36 39	64 66		92	
17. Cyanide, total,[14] milligrams per liter.	Distillation followed by silver nitrate titration or pyridine pyrazolone (or barbituric acid) colorimetric.	40	361	503	85	[10](22)
18. Cyanide amenable to chlorination, milligrams per liter.	do	40	370	505		
19. Dissolved oxygen, milligrams per liter.	Winkler (Azide modification) or electrode method.	51 56	443 450	368	126	[4](600)
20. Fluoride, milligrams per liter.	Distillation [4] followed by ion electrode; SPADNS; or automated complexone.	 65 59 61	389 391 393 614	 307 305	 163	
21. Hardness—Total, as $CaCO_3$, milligrams per liter.	EDTA titration; automated colorimetric; or atomic absorption (sum of Ca and Mg as their respective carbonates).	68 70	202	161	94	[4](617)
22. Hydrogen ion (pH), pH units.	Electrometric measurement.	239	460	178	129	[4](608)
23. Kjeldahl nitrogen (as N), milligrams per liter.	Digestion and distillation followed by nesslerization, titration, or electrode; automated digestion automated phenolate.	175 165 182	437		122	[4](612)
METALS						
24. Aluminum—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Eriochrome Cyanine R).	92	152 171		[11](19)	
25. Aluminum—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced methods for total aluminum.					
26. Antimony—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]	94				
27. Antimony—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total antimony.					
28. Arsenic—Total, milligrams per liter.	Digestion followed by silver diethyldithiocarbamate; or atomic absorption.[16] [18]	 9 95	283 [illegible] 159		 [10](31) [18](37)	
29. Arsenic—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total arsenic.					
30. Barium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]	97	152		32	
31. Barium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total barium.					
32. Beryllium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Aluminon).	99	152 177		53	
33. Beryllium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total beryllium.					
34. Boron—Total, milligrams per liter.	Colorimetric (Curcumin).	13	287			
35. Boron—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total boron.					
36. Cadmium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Dithizone).	101	148 182	345	62	[8](619) [10](37)
37. Cadmium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total cadmium.					
38. Calcium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption; or EDTA titration.	103	148 189	345	66	
39. Calcium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total calcium.					
40. Chromium VI, milligrams per liter.	Extraction and atomic absorption; colorimetric (Diphenylcarbazide).	89, 105	 192		[9]78 75	
41. Chromium VI—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for chromium VI.					
42. Chromium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Diphenylcarbazide).	105	148 192	343 286	75 77	[8](619)
43. Chromium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total chromium.					

See footnotes at end of table.

Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.) Pt. 31 1975 ASTM	USGS methods [3]	Other approved methods
44. Cobalt—Total, milligrams per liter.	Digestion [14] followed by atomic absorption.[16]	107	148	345	80	[19] (27)
45. Cobalt—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total cobalt.					
46. Copper—Total, milligrams per liter.	Digestion [14] followed by atomic absorption [16] or by colorimetric (Neocuproine).	108	148 196	345 293	82	[3] (619) [19] (37)
47. Copper—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total copper.					
48. Gold—Total, milligrams per liter.	Digestion [14] followed by atomic absorption.[19]					
49. Iridium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption.[19]					
50. Iron—Total, milligrams per liter.	Digestion [14] followed by atomic absorption [16] or by colorimetric (Phenanthroline).	110	148 208	345 286	102	[3] (619)
51. Iron—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total iron.					
52. Lead—Total, milligrams per liter.	Digestion [14] followed by atomic absorption [16] or by colorimetric (Dithizone).	112	148 215	345	105	[3] (619)
53. Lead—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total lead.					
54. Magnesium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption; or gravimetric.	114	148 221	345	109	[3] (619)
55. Magnesium—Dissolved milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total magnesium.					
56. Manganese—Total milligrams per liter.	Digestion [14] followed by atomic absorption [16] or by colorimetric (Persulfate or periodate).	116	148 225, 227	345	111	[3] (619)
57. Manganese—Dissolved milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total manganese.					
58. Mercury—Total, milligrams per liter.	Flameless atomic absorption.	118	156	[illegible]	[20] (51)	
59. Mercury—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total mercury.					
60. Molybdenum—Total, milligrams per liter.	Digestion [14] followed by atomic absorption.[16]	139		350		
61. Molybdenum—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total molybdenum.					
62. Nickel—Total, milligrams per liter.	Digestion [14] followed by atomic absorption [16] or by colorimetric (Heptoxime).	141	148	345	115	
63. Nickel—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total nickel.					
64. Osmium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption.[19]					
65. Palladium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption.[19]					
66. Platinum—Total, milligrams per liter.	Digestion [14] followed by atomic absorption.[19]					
67. Potassium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption, colorimetric (Cobaltinitrite), or by flame photometric.	143	285 284	403	134	[3] ([illegible])
68. Potassium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total potassium.					
69. Rhodium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption.[19]					
70. Ruthenium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption.[19]					
71. Selenium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption.[16] [19]	145	159			
72. Selenium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total selenium.					
73. Silica—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by colorimetric (Molybdosilicate).	274	487	398	139	
74. Silver—Total,[20] milligrams per liter.	Digestion [14] followed by atomic absorption [16] or by colorimetric (Dithizone).	146	148 243		142	[3](619) [19](37)
75. Silver—Dissolved,[20] milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total silver.					
76. Sodium—Total, milligrams per liter.	Digestion [14] followed by atomic absorption or by flame photometric.	147	250	403	148	[3] ([illegible])
77. Sodium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total sodium.					

See footnotes at end of table

Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.)		Other approved methods
				Pt. 31 1975 ASTM	USGS methods [2]	
78. Thallium—Total, milligrams per liter.	Digestion [16] followed by atomic absorption.[16]	149				
79. Thallium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total thallium.					
80. Tin—Total, milligrams per liter.	Digestion [16] followed by atomic absorption.[16]	150			[11] (65)	
81. Tin—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total tin.					
82. Titanium—Total, milligrams per liter.	Digestion [16] followed by atomic absorption.[16]	151				
83. Titanium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total titanium.					
84. Vanadium—Total, milligrams per liter.	Digestion [16] followed by atomic absorption [16] or by colorimetric (Gallic acid).	153	152 260	 661	 [11] (67)	
85. Vanadium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total vanadium.					
86. Zinc—Total, milligrams per liter.	Digestion [16] followed by atomic absorption [16] or by colorimetric (Dithizone).	155	148 265	345	150	[3](619) [10](37)
87. Zinc—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total zinc.					
88. Nitrate (as N), milligrams per liter.	Cadmium reduction; brucine sulfate; automated cadmium or hydrazine reduction.[21]	201 197 207	423 427 620	 358	 119	 [3](614) [10](28)
89. Nitrite (as N), milligrams per liter.	Manual or automated colorimetric (Diazotization).	215	434		121	
90. Oil and grease, milligrams per liter.	Liquid-liquid extraction with trichloro-trifluoroethane-gravimetric.	229	515			
91. Organic carbon; total (TOC), milligrams per liter.	Combustion—Infrared method.[22]	236	532	467	[10] (4)	
92. Organic nitrogen (as N), milligrams per liter.	Kjeldahl nitrogen minus ammonia nitrogen.	175, 159	437		122	[3] (612, 614)
93. Orthophosphate (as P), milligrams per liter.	Manual or automated ascorbic acid reduction.	249 256	481 624	384	131	[3] (621)
94. Pentachlorophenol, milligrams per liter.	Gas chromatography [18].					
95. Pesticides, milligrams per liter.	do.[19]		555	320	[20] (24)	
96. Phenols, milligrams per liter.	Colorimetric, (4AAP)	241	582	545		
97. Phosphorus (elemental), milligrams per liter.	Gas chromatography [20].					
98. Phosphorus; total (as P), milligrams per liter.	Persulfate digestion followed by manual or automated ascorbic acid reduction.	249 256	476, 481 624	384	133	[3] (621)
RADIOLOGICAL						
99. Alpha—Total, pCi per liter	Proportional or scintillation counter.		648	591 [11]	[20] (75+78)	
100. Alpha—Counting error, pCi per liter.	do.		648	594	[20] (79)	
101. Beta—Total, pCi per liter	Proportional counter		648	598 [11]	[20] (75+78)	
102. Beta—Counting error, pCi per liter.	do.		648	598	[20] (79)	
103. (a) Radium—Total, pCi per liter.	do.		661	661		
(b) ^{226}Ra, pCi per liter	Scintillation counter		667		[20] (81)	
RESIDUE						
104. Total, milligrams per liter	Gravimetric, 103 to 105° C.	270	91			
105. Total dissolved (filterable), milligrams per liter.	Glass fiber filtration, 180° C.	266	92			
106. Total suspended (nonfilterable), milligrams per liter.	Glass fiber filtration, 103 to 105° C.	268	94			
107. Settleable, milliliters per liter or milligrams per liter.	Volumetric or gravimetric		95			
108. Total volatile, milligrams per liter.	Gravimetric, 550° C.	272	95			
109. Specific conductance, micromhos per centimeter at 25° C.	Wheatstone bridge conductimetry.	275	71	120	148	[3] (606)
110. Sulfate (as SO_4), milligrams per liter.	Gravimetric; turbidimetric; or automated colorimetric (barium chloranilate).	 277 279	493 496	424 425		[3] (624) [3] (623)
111. Sulfide (as S), milligrams per liter.	Titrimetric—Iodine for levels greater than 1 mg per liter; Methylene blue photometric.	284	505 503		154	
112. Sulfite (as SO_3), milligrams per liter.	Titrimetric, iodine-iodate	285	508	435		
113. Surfactants, milligrams per liter.	Colorimetric (Methylene blue).	157	600	494	[20] (11)	
114. Temperature, degrees C	Calibrated glass or electrometric thermometer.	286	125		[20] (21)	
115. Turbidity, NTU	Nephelometric	295	132	223	156	

---

[1] Recommendations for sampling and preservation of samples according to parameter measured may be found in "Methods for Chemical Analysis of Water and Wastes, 1974" U.S. Environmental Protection Agency, table 2, pp. viii-xii.

[2] All page references for USGS methods, unless otherwise noted, are to Brown, E., Skougstad, M. W., and Fishman, M. J., "Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases," U.S. Geological Survey Techniques of Water-Resources Inv., book 5, ch. A1, (1970).

[3] EPA comparable method may be found on indicated page of "Official Methods of Analysis of the Association of Official Analytical Chemists" methods manual, 12th ed. (1975).

[4] Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.

[5] The method used must be specified.

[6] The 5 tube MPN is used.

[7] Slack, K. V. and others, "Methods for Collection and Analysis of Aquatic Biological and Mircobiological Samples: U.S. Geological Survey Techniques of Water-Resources Inv. book 5, ch. A4 (1973)."

[8] Since the membrane filter technique usually yields low and variable recovery from chlorinated wastewaters, the MPN method will be required to resolve any controversies.

[9] Adequately tested methods for benzidine are not available. Until approved methods are available, the following interim method can be used for the estimation of benzidine: (1) "Method for Benzidine and Its Salts in Wastewaters," available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[10] American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, N.Y. 10018.

[11] Fishman, M. J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey for Analysis of Wastewaters," (1976) open-file report 76-177.

[12] Procedures for pentachlorophenol, chlorinated organic compounds ,and pesticides can be obtained from the Environmental Monitoring and Support Lbaoratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[13] Color method (ADMI procedure) available from Environmental Monitoring and Support Lbaoratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[14] For samples suspected of having thiocyanate interference, magnesium chloride is used as the digestion catalyst. In the approved test procedure for cyanides, the recommended catalysts are replaced with 20 ml of a solution of 510 g/l magnesium chloride ($MgCl_2 \cdot 6H_2O$). This substitution will eliminate thiocyanate interference for both total cyanide and cyanide amendable to chlorination measurements.

[15] For the determination of total metals the sample is not filtered before processing. Because vigorous digestion procedures may result in a loss of certain metals through preciptation, a less vigorous treatment is recommended as given on p. 83 (4.1.4) of "Methods for Chemical Analysis of Water and Wastes" (1974). In those instances where a more vigorous digestion is desired the procedure on p. 82 (4.1.3) should be followed. For the measurement of the noble metal series (gold, iridium, osmium, palladium, platinum, rhodium and ruthenium), an aqua regia digestion is to be substituted as follows: Transfer a representitive aliquot of the well-mixed sample to a Griffin beaker and add 3 ml of concentrated redistilled HNO_3. Place the beaker on a steam bath and evaporate to dryness. Cool the beaker and cautiously add a 5 ml portion of aqua regia. (Aqua regia is prepared immediately before use by carefully adding 3 volumes of concentrated HCl to one volume of concentrated HNO_3.) Cover the beaker with a watch glass and return to the steam bath. Continue heating the covered beaker for 50 min. Remove cover and evaporate to dryness. Cool and take up the residue in a small quantity of 1:1 HCl. Wash down the beaker walls and watch glass with distilled water and filter the sample to remove silicates and other insoluble material that could clog the atomizer. Adjust the volume to some predetermined value based on the expected metal concentration. The sample is now ready for analysis.

[16] As the various furnace devices (flameless AA) are essentially atomic absorption techniques, they are considered to be approved test methods. Methods of standard addition are to be followed as noted in p. 78 of "Methods for Chemical Analysis of Water and Wastes," 1974.

[17] Dissolved metals are defined as those constituents which will pass though a 0.45 μm membrane filter. A prefiltration is permissible to free the sample from larger suspended solids. Filter the sample as soon as practical after collection using the first 50 to 100 ml to rinse the filter flask. (Glass or plastic filtering apparatus are recommended to avoid possible contamination.) Discard the portion used to rinse the flask and collect the required volume of filtrate. Acidify the filtrate with 1:1 redistilled HNO_3 to a pH of 2. Normally, 3 ml of (1:1) acid per liter should be sufficient to preserve the samples.

[18] See "Atomic Absorption Newsletter," vol. 13, 75 (1974). Available from Perkin-Elmer Corp., Main Ave., Norwalk, Conn. 06852.

[19] Method available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[20] Recommended methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/l and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/l 20 ml of sample should be diluted to 100 ml by adding 40 ml each of 2M $Na_2S_2O_3$ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/l the recommended method is satisfactory.

[21] An automated hydrazine reduction method is available from the Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[22] A number of such systems manufactured by various companies are considered to be comparable in their performance. In addition, another technique, based on combustion-methane detection is also acceptable.

[23] Goerlitz, D., Brown, E., "Methods for Analysis of Organic Substances in Water": U.S. Geological Survey Techniques of Water-Resources Inv., book 5, ch. A3 (1972).

[24] R. F. Addison and R. G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," "Journal of Chromatography," vol. 47, No. 3, pp. 421-426, 1970.

[25] The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only suspended. Therefore, the 2 results must be added together to obtain "total."

[26] Stevens, H. H., Ficke, J. F., and Smoot, G. F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation: U.S. Geological Survey Techniques of Water Resources Inv., book 1 (1975)."

4. In § 136.4, the second sentence of paragraph (c) is amended by deleting the word "subchapter" immediately following the phrase "procedure under this" and immediately preceding the word "shall" and replaced with the phrase "paragraph c;" and § 136.4 is amended by adding a new paragraph (d) to read as follows:

§ 136.4 Application for alternate test procedures.

* * * * *

(c) * * * Any application for an alternate test procedure under this paragraph (c) shall: * * *

(d) An application for approval of an alternate test procedure for nationwide use may be made by letter in triplicate to the Director, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268. Any application for an alternate test procedure under this paragraph (d) shall:

(1) Provide the name and address of the responsible person or firm making the application.

(2) Identify the pollutant(s) or parameter(s) for which nationwide approval of an alternate testing procedure is being requested.

(3) Provide a detailed description of the proposed alternate procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure to the pollutant(s) or parameter(s) in waste water discharges from representative and specified industrial or other categories.

(4) Provide comparability data for the performance of the proposed alternate test procedure compared to the performance of the approved test procedures.

§ 136.5 [Amended]

5. In § 136.5, paragraph (a) is amended by inserting the phrase "proposed by the responsible person or firm making the discharge" immediately after the words "test procedure" and before the period that ends the paragraph.

6. In § 136.5, paragraph (b) is amended by inserting in the first sentence the phrase "proposed by the responsible person or firm making the discharge" immediately after the words "such application" and immediately before the comma. The second sentence of paragraph (b) is amended by deleting the phrase "Methods Development and Quality Assurance Research Laboratory" immediately after the phrase "State Permit Program and to the Director of the" at the end of the sentence, and inserting in its place the phrase "Environmental Monitoring and Support Laboratory, Cincinnati."

7. In § 136.5, paragraph (c) is amended by inserting the phrase "proposed by the responsible person or firm making the discharge" immediately after the phrase "application for an alternate test procedure" and immediately before the comma; and by deleting the phrase "Methods Development and Quality Assurance Research Laboratory" immediately after the phrase "application to the Director of the" and immediately before the phrase "for review and recommendation" and inserting in its place the phrase "Environmental Monitoring and Support Laboratory, Cincinnati."

8. In § 136.5, the first sentence of paragraph (d) is amended by inserting the phrase, "proposed by the responsible person or firm making the discharge," immediately after the phrase, "application for an alternate test procedure," and immediately before the comma.

The second sentence of paragraph (d) is amended by deleting the phrase, "Methods Development and Quality Assurance Research Laboratory," immediately after the phrase, "to the Regional Administrator by the Director of the," and immediately preceding the period ending the sentence and inserting in its place the phrase, "Environmental Monitoring and Support Laboratory, Cincinnati."

The third sentence of paragraph (d) is amended by deleting the phrase, "Methods Development and Quality Assurance Research Laboratory," immediately after the phrase, "forwarded to the Director," and immediately before the second comma and by inserting in its place the phrase, "Environmental Monitoring and Support Laboratory, Cincinnati."

9. Section 136.5 is amended by the addition of a new paragraph (e) to read as follows:

§ 136.5 Approval of alternate test procedures.

* * * * *

(e) Within ninety days of the receipt by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati of an application for an alternate test procedure for nationwide use, the Director of the Environmental Monitoring and Support Laboratory, Cincinnati shall notify the applicant of his recommendation to the Administrator to approve or reject the application, or shall specify additional information which is required to determine whether to approve the proposed test procedure. After such notification, an alternate method determined by the Administrator to satisfy the applicable requirements of this part shall be approved for nationwide use to satisfy the requirements of this subchapter; alternate test procedures determined by the Administrator not to meet the applicable requirements of this part shall be rejected. Notice of these determinations shall be submitted for publication in the Federal Register not later than 15 days after such notification and determination is made.

[FR Doc.76-35082 Filed 11-30-76;8:45 am]

2. Announcing the Course

a. Course Availability

Course availability is most likely to be established through one of two mechanisms:

1) Management or regulatory authority determines that training is required, makes arrangements for course presentation and instructs designated personnel to appear at a specified time and place for training; or,

2) The course may be planned by a training organization which schedules and publicly announces the course either as a special offering or as an element of an overall curriculum of training.

b. Responsibility for Course Announcement

All training organizations should establish and maintain mailing lists of officials, organizations and interested individuals to whom training announcements should be addressed.

Course announcements should be released by the training organization and/or the sponsoring agency (if applicable).

When a special course offering is planned at the request of management or regulatory authority with identified class participants, it usually is best for the requesting authority to make the announcement.

c. Types of Course Announcements

1) Training bulletins, or catalogues are widely used by established training organizations, and should be used for announcement of this course when offered as part of an on-going curriculum of courses.

2) Special fliers or brochures should be developed for public announcement through established mailing lists. These releases may be used for regular offerings of an on-going curriculum of courses; but they are particularly applicable when a special offering of the course is planned.

3) The course may be announced in a journal, newsletter or other periodical widely read by the personnel for whom the training is intended.

4) The course may be announced by personal letter or other direct communication with a student assigned to take the training.

d. Timing of Course Announcements

Training catalogues or bulletins usually are for a period of one year or more. Accordingly, the prospective student should have from three months to one year of advance notice of the training.

When the course is a special offering announced through a flier or other special mechanism, at least 90 days should be provided between the release of the announcement and the start of the course. There are at least two reasons for this:

1) Permits course applicants to secure necessary approvals for attendance, and to make personal scheduling arrangements; and,

2) Provides course presentation staff with lead time for course preparations, acquiring special instructional materials, preparation of laboratory supplies and equipment, and related tasks.

e. Information Provided in Course Announcements

The following list should be helpful as a checklist to those preparing a course announcement. Samples of an announcement for this course as it might appear in a catalogue of courses, and as it might appear in a special flier are shown in the section of this Guide titled SECRETARIAL SUPPORT.

In the event that the course is announced in a periodical, the editor may apply constraints on style and format which make it impossible to provide all the pertinent information on the course. In such cases the announcement must provide the name and address of an office from which further information can be obtained. The information to be provided should be as complete as that given in a course catalogue or flier and, naturally, should include any additional special information specifically requested.

The following will be helpful as a checklist to those preparing an original course announcement:

1) Course title, dates and location

2) Name of organization conducting the course (and name of co-sponsor, if applicable)

3) Description of intended student body, reason why this training is needed and summary of Course content.

4) Prerequisites for attendance (special skills or training which the applicant must have for admission)

5) Description of the training environment to be used (classroom, laboratory, field, in-plant, etc.)

6) Identification of knowledge and skills the participant will have on satisfactory completion of training

7) Tuition (if applicable)

8) How and where to apply for admission to this course

3. Summary Plan for the Course

A convenient format to use in the early stages of devising a course plan is a day-to-day assignment of time blocks based on estimates by authors of the training time required for each parameter. (An example is on the next page.) Using available time as a first criterion will allow a variety of possible sequences. Then other considerations should be applied. Some examples are:

a. If some equipment must be used in more than one test, schedule another topic between the two tests to allow time for the required clean-up.

b. Schedule the topics so each instructor alternates between prime and assistant responsibilities to allow time for preparations which must be done right before training sessions.

c. If one procedure requires skills taught in another procedure, order the presentations accordingly.

d. If most students need only 80% of the procedures taught, schedule the remaining 20% of the procedures as a group so students can conveniently schedule their attendance for training pertinent to their needs.

EXAMPLE - Summary Plan for the Course:

ESTIMATED AGENDA FOR COURSE 164.3

MONDAY		TUESDAY		WEDNESDAY		THURSDAY		FRIDAY	
Activity	Time Hours	Activity	Time Hours	Activity	Time Hours	Activity	Time Hours	Activity	Time Hours
Registration	1/4	Evaluation	1/2	Evaluation TKN	1/2	Titrations	1/2	Evaluation P	1/2
Introductions	1/4	Total Nitrogen and Organic Nitrogen	3-1/2	Evaluation NH_3	1/2	Chemical Oxygen Demand	3-1/2	Reporting Data	1/2
Course Objectives	1/2			Nitrate and Nitrite	3			Oil and Grease	2-1/2
Permit Requirements	1/2								
Sampling and Preservation	1/2					Evaluation COD	1/2	Course Closing Exercises	1/2
Lunch	1	Lunch	1	Lunch	1	Lunch	1		
Lab/Safety Orientation	3/4	Total Nitrogen and Organic Nitrogen (cont.)	1-1/4	Nitrate and Nitrite	1	Phosphorus	3-1/2		
Use of Spec 20	2								
Constructing Calibration Curves	1	Ammonia	2	Nitrate	1-1/2				
Lab Exercise?				Evaluation NO_3 NO_2	1/2				
TOTAL	6-3/4	TOTAL	8-1/4	TOTAL	8	TOTAL	9	TOTAL	4

NOTES:

1. Teaching time is available Monday. Thursday is a long day.
2. Phosphorus glassware requires special cleaning and is used for other tests. Schedule this earlier in week.
3. Use of Spec 20 and Calibration Curves must be taught before Total Nitrogen, Nitrate and Nitrite, and Phosphorus.
4. COD can be taught in two segments, oxidation in the afternoon. titrations the next morning.
5. Keep Oil and Grease on Friday. A different test could be taught then if local needs suggest it.

4. Sample Course Schedule

EFFLUENT MONITORING PROCEDURES:
NUTRIENTS (164.3)

(Location)
(Date)

Course Coordinator:

DAY AND TIME	SUBJECT	OUTLINE	INSTRUCTOR*
Monday			
8:30 - 8:50	Registration - Introductions		Course Coordinator
8:50 - 9:30	NPDES Requirements Course Objectives EMP Format		Course Coordinator
9:30 - 9:45	Laboratory & Safety Orientation		Chemist #1
9:45 - 11:15	Use of a Spectrophotometer	1	Chemist #2 (Chemist #1)
11:15 - 12:15	Preparation of Calibration Graphs	2	Chemist #2 (Chemist #1)
12:15 - 1:15	Lunch		
1:15 - 2:00	Determination of Total Phosphorus	3	Chemist #3 (Chemist #2)
2:00 - 3:30	Determination of Total Phosphorus: Glassware Preparation	3	Chemist #3 (Chemist #2)
3:30 - 5:00	Determination of Total Phosphorus: Preparation of Standard	3	Chemist #3 (Chemist #2)
Tuesday			
8:30 - 12:00	Determination of Total Phosphorus	3	Chemist #3 (Chemist #2)
12:00 - 1:00	Lunch		
1:00 - 1:45	Determination of Total Phosphorus (Concluded)	3	Chemist #3 (Chemist #2)
1:45 - 4:30	Determination of Chemical Oxygen Demand	4	Chemist #1 (Chemist #3)
Wednesday			
8:30 - 10:00	Determination of Chemical Oxygen Demand	4	Chemist #1 (Chemist #3)
10:00 - 10:15	Break		
10:15 - 12:00	Determination of Total Kjeldahl Nitrogen	5	Chemist #2 (Chemist #1)

DAY AND TIME	SUBJECT	OUTLINE	INSTRUCTOR*
Wednesday (Continued)			
12:00 - 1:00	Lunch		
1:00 - 3:00	Determination of Total Kjeldahl Nitrogen, Micro Distillation -Nesslerization and/or -Selective Ion Electrode	5 9	Chemist #2 (Chemist #1)
3:00 - 5:00	Determination of Ammonia Nitrogen, Macro Distillation -Titration	6	Chemist #3 (Chemist #2)
Thursday			
8:00 - 8:15	Determination of Chemical Oxygen Demand (Concluded)	4	Chemist #1 (Chemist #3)
8:15 - 12:15	Determination of Nitrate and Nitrite Nitrogen	7	Chemist #1 (Chemist #3)
12:15 - 1:15	Lunch		
1:15 - 5:00	Determination of Nitrate and Nitrite Nitrogen (Concluded)	7	Chemist #1 (Chemist #3)
Friday			
8:30 - 11:30	Determination of Oil and Grease	8	Chemist #2 (Chemist #1)
11:30 - 12:15	Reporting Self-Monitoring Data		Chemist #3 (Chemist #2)
12:15 - 12:30	Course Closing		Course Coordinator

*NOTE: For actual course presentation, insert the name of the instructor instead of the technical specialty, as shown.

5. Milestones in Course Planning and Preparation

The following pages list major areas of course responsibilities in a chronological order to facilitate orderly and timely accomplishment. The table also serves as an example for assignment of these responsibilities to various staff members. The Staff Guide (Chapter) number of the related outline is given for each listing so that the user can quickly find the details about his/her assignments.

The table headings are job titles associated with the listed tasks. A maximum staff is cited, including a laboratory assistant. It is recognized, however, that staff is often limited and one individual may serve in several of the defined roles. Having this summary according to an ideal situation should facilitate an equitable division of the required tasks among fewer persons.

About three months before the course, decisions must be made regarding specific procedures to be taught, because several of the Effluent Monitoring Procedures (EMPs) contain a choice of procedures.

The ammonia EMP is written for the distillation of a macro sample followed by either Nesslerization or titration. The Kjeldahl Nitrogen EMP contains a choice between macro or micro digestion and distillation followed by either Nesslerization or titration. There is also an EMP for determining ammonia by using a selective ion electrode. The Chemical Oxygen Demand EMP is written for the low level range of 5-50 mg/liter, but includes the modifications for concentrations greater than 50 mg/liter COD. The Phosphorus EMP can be used to determine total phosphorus or only orthophosphate. These different possibilities affect the equipment preparations needed prior to the course.

The requirements of area NPDES permits and/or the type of equipment readily available will help the course planners make choices among these possibilities.

	Training Supervisor	Course Coordinator	Course Secretary	Chemist #1	Chemist #2	Chemist #3	Lab Assistant	Staff Guide Outline No.
5 TO 6 MONTHS BEFORE COURSE								
Determination of the need and decision to have course.	x							1, 2
Designation of Course Director and Course Secretary.	x							
Review responsibilities.		x						PREFACE, 1 thru 12
Review responsibilities.			x					PREFACE, 1, 6, 10, 11, 12
Commit classroom and laboratory facilities.	x	x						7
Develop and release Course Announcement including location, date, general statement of course content and training objectives.		x	x					2
Prepare all forms and information sheets related to student registration procedures.		x	x					10, 11, 12
Decide on staff members.	x	x						6
4 TO 5 MONTHS BEFORE COURSE								
Receive, review, act upon Course Applications, continuing until course begins.		x	x					10, 11, 12
Maintain records on deposition of each application, continuing through course.			x					10, 11, 12
Inventory Staff Guides, Order needs.		x	x					9

	Training Supervisor	Course Coordinator	Course Secretary	Chemist #1	Chemist #2	Chemist #3	Lab Assistant	Staff Guide Outline No.
3 MONTHS BEFORE COURSE								
Commit all staff members who will participate in Course.	x	x		x	x	x	x	6
Develop Milestone Checklist for Course.		x	x					5
Distribute copies to staff of Milestones, Staff Guide, Student Text and any other pertinent training resources.	x	x	x					5, 9
Review responsibilities.				x	x	x	x	PREFACE, 1, 6, 10, 12, 13
Assign topics to Primary (P) and Assistant (A) Instructors:	x	x						
Spectrophotometer				A	P			14
Calibration Graphs				A	P			15
Phosphorus					A	P		16
Chemical Oxygen Demand				P		A		17
Kjeldahl Nitrogen				A	P			18
Ammonia					A	P		19
Ammonia Electrode					A	P		22
Nitrate-Nitrite (Cd)				P		A		20
Oil and Grease				A	P			21
Decide which procedures to teach if EMP has choices. Record on related IPW.	x	x		x	x	x	x	8 and 13 thru 22
Mark which procedures are to be taught on the summary of laboratory equipment and supply needs for course.		x		x	x	x	x	8
Develop summary plan for course.		x	x	x	x	x	x	3
Inventory chemicals and laboratory equipment/supplies. List and commit lending sources. Order rest of needs.		x	x	x	x	x	x	8
Inventory classroom equipment/supplies. List and commit lending sources. Order rest of needs.		x	x					7

(Continued)

	Training Supervisor	Course Coordinator	Course Secretary	Chemist #1	Chemist #2	Chemist #3	Lab Assistant	Staff Guide Outline No.
Inventory student reference texts. Order needs.		x	x					9
2 MONTHS BEFORE COURSE								
Finalize Course Schedule (Agenda).		x	x	x	x	x	x	4
Request laboratory/classroom needs from lending sources.		x	x	x	x	x	x	7, 8
Request training aids from lending sources.		x	x	x	x	x		9
6 WEEKS BEFORE COURSE								
Check out operation of all items listed as "A. Capital Equipment" plus COD reflux apparatus.				x	x	x	x	8
Primary and Assistant Instructors go through EMP laboratory procedures in student reference texts, using IPWs to standardize instructions for students.				x	x	x		13 thru 22
1 MONTH BEFORE COURSE								
Summary (to date) to staff of registered students, continuing to course beginning.		x	x					
Check on progress of staff preparations for instruction, continuing through course.		x		x	x	x		6
Prepare all administrative forms and materials needed for course presentation.		x	x					10, 11, 12
Plan and rehearse classroom presentations using all required training aids. Finalize.				x	x	x		13 thru 22

(Continued)

	Training Supervisor	Course Coordinator	Course Secretary	Chemist #1	Chemist #2	Chemist #3	Lab Assistant	Staff Guide Outline No.
Obtain any duplicated instructional materials (data sheets, etc.).			x	x	x	x	x	12 and 13 thru 22
Review summary of laboratory equipment and supply needs for expected number of students doing the selected procedures.		x					x	8
Clean all glassware required by students. [(Special for Phosphorus, COD, Ammonia (distillation apparatus)]							x	8, 16, 17, 18, 19
Reserve all specially-cleaned glassware.							x	8, 16, 17, 18, 19
Assemble other student equipment and supplies.							x	8
2 WEEKS BEFORE COURSE								
Arrange for security of classroom and laboratory.		x	x					7
Make reagents required by students EXCEPT those with specified, limited stability.				x	x	x	x	8 and 13 thru 22
Make final arrangements to obtain required effluent samples.				x	x	x	x	8 and 13 thru 22
Determine range of concentration of desired constituent in effluent sample from source of course samples.				x	x	x		8 and 13 thru 22
Arrange for disposal of special test wastes (COD, alkaline wastes, Cd)				x	x		x	17, 18, 19, 20

(Continued)

	Training Supervisor	Course Coordinator	Course Secretary	Chemist #1	Chemist #2	Chemist #3	Lab Assistant	Staff Guide Outline No.
Give Laboratory Assistant final list of equipment and supplies to be at each laboratory position. Discuss arrangement of shared equipment.				x	x	x		13 thru 22
1 WEEK BEFORE COURSE								
Inform building food service of number of expected students and course lunch times (as appropriate).			x					
3 DAYS BEFORE COURSE								
Finalize seating arrangement for classroom.		x						7
Assemble course materials in classroom (student texts, administrative materials, etc.). Distribute as appropriate.		x	x					9, 10, 11, 12
Ready classroom instructional aids (boards, erasers, etc.)		x	x				x	7
Check out all classroom equipment (electrical systems, PA, projection equipment) and obtain back-up accessories (bulbs, etc.).		x						7
COURSE OPENING								
Conduct opening exercises.		x						6
Participate in course opening.	x		x	x	x	x	x	6
Complete any required student records, including roster.		x	x					10, 11, 12
(Continued)								

	Training Supervisor	Course Coordinator	Course Secretary	Chemist #1	Chemist #2	Chemist #3	Lab Assistant	Staff Guide Outline No.
Prepare course certificates and give to Day's Instructor.			x					10, 12
EVERY DAY OF COURSE								
Maintain general supervision of course.		x						6
Prepare unstable reagents and/or samples on day of test.				x	x	x	x	8, 16, 18, 19, 20, 22
Obtain effluent samples for each test on day of test.				x	x	x	x	8, 16, 17, 18, 19, 21
When assistant instructor, make any student evaluation records requested by the lead instructor.				x	x	x		1, 6, 10, 13 thru 22
When primary instructor, compile evaluation record for each student.				x	x	x		1, 6, 10, 13 thru 22
When primary instructor, sign certificate of each student who satisfactorily performs test.				x	x	x		1, 12, 13 thru 22
After signing certificates, give them to next primary instructor.				x	x	x		
When primary instructor, make arrangements to work with each non-qualifying student.				x	x	x		1, 13 thru 22
Oversee disposal of special test wastes.				x	x		x	17, 18, 19, 20

	Training Supervisor	Course Coordinator	Course Secretary	Chemist #1	Chemist #2	Chemist #3	Lab Assistant	Staff Guide Outline No.
SECOND-LAST DAY OF COURSE								
Distribute course critique sheet to students.		x						
LAST DAY OF COURSE								
Assemble certificates, check for completeness and sign.		x						12
Collect students' critique sheets.		x						
Conduct closing exercises and distribute certificates.		x						6
Participate in course closing.	x		x	x	x	x	x	6
Clean up classroom and laboratory.		x	x	x	x	x	x	6
WITHIN A WEEK OF COURSE PRESENTATION								
Return or replace any borrowed classroom equipment/ supplies.		x	x					7
Return or replace any borrowed laboratory equipment/ supplies.				x	x	x	x	8
Return or replace any borrowed training aids.		x	x	x	x	x		9
Order repairs or replacements of own equipment used in course.		x	x	x	x	x		7, 8, 9
File evaluation records on all student in predetermined area.			x	x	x	x		10

(Continued)

	Training Supervisor	Course Coordinator	Course Secretary	Chemist #1	Chemist #2	Chemist #3	Lab Assistant	Staff Guide Outline No.
Participate in staff session on evaluation of course and recommendations for future offerings.	x	x	x	x	x	x	x	6, 10
Prepare course summary/ evaluation report.		x						6
Complete and file entire course records in mutually determined area.		x	x					10, 11, 12

B. Training Resources

This section considers four topics: Training Staff, Training Facilities, Laboratory Equipment and Supply Requirements, and Instructional Resources.

Staffing plans, facilities, equipment and supplies described in this Guide are based on a class of 18 students. For at least the first several (3 or 4) course offerings, it is urged that this maximum number of students per class be strictly adhered to.

A staff experienced in presentation of this course may be able to increase class size to, say, 24 students. On no account should this laboratory-oriented course exceed 24 students with an instructional staff of the size described here. Large numbers of students per instructor simply cannot be provided with the individual attention and instruction required for effective training and assurance that they have indeed learned to perform the analyses which are the subject of this course.

If the number of workers requiring this course is quite large, the best course of action is to meet the training need through providing a greater number of course offerings.

The required instructional resources, shown in outline 9, are compiled from the individual instructional package worksheets shown in Part II. Training administrators should coordinate requests for instructional resources to be acquired, through loan or through purchase, from other sources. This outline provides a basis for such coordination.

1. Training Staff

Each member of the training staff for this, as for any short course, is a member of a team. This team can function effectively only through each member's understanding of the training goals to be met and the plans for meeting these goals, through performance of his/her own duties, and through providing mutually supportive activity with other team members for the effective conduct of the course as a whole.

a. Qualifications of Instructional Staff

1) Each should have a thorough knowledge of the subject matter for which he/she has responsibility, including a high order of technical skill in any procedures to be carried out.

2) Each should be able to perform effectively as an instructor, both in the classroom and in the laboratory. This includes ability to make rapid adjustments in the style and technical level of instruction in order to work with students having a varied range of entry-level knowledge, skills, and prior education.

3) Each should be willing to accept a certain rigidity in the choice of analytical procedures to be taught, in accordance with policies

and formal directives of the applicable regulatory authority (-ies). The basis for, and recommended procedures to be followed, in introducing variations in methods to be taught in tests and measurements of municipal effluents is discussed elsewhere in this Guide.

b. Estimated Time Allocations for Training Staff

Each member of the training staff has specific duties before, during, and after the scheduled course dates. For planning purposes, it is assumed that pre-course activity will begin three months or more, as required, before classroom instruction begins. During this period, the estimated time allocations will permit the phasing-in of work activity for this course with other duties of all personnel. During the course, all instructional staff and laboratory assistant (if used) are fully occupied, and should not be given any other duty assignments. Post-course activities should be completed within one or two weeks after the last day of instruction. In the post-course period, all staff may begin to phase in other duties pending final completion of all details associated with this training effort.

Staff Identification	Working Days (estimate)		
	Before	During	After
Course Administration			
Course Coordinator (ordinarily, this is one of the instructional staff, who is assigned double-duty as coordinator-instructor)	12	1	2
Course Secretary	10	2	2
Instructional Staff			
Chemist #1	8	5	2
Chemist #2	8	5	2
Chemist #3	8	5	2
Laboratory Support			
Laboratory Assistant (optional)	10	5	5

c. Duties and Responsibilities of Training Staff

1) Course Coordinator

This individual may be known variously as Course Director, Course Leader, or by some other term suitable to the situation. In most cases the Course Coordinator will function in a dual capacity as one of the instructional staff for the course. In principle, any one of the Instructors could function as Course Coordinator. In the absence of other factors, it may be best to have the individual who bears the lightest teaching load to act as Course Coordinator.

a) Before the course, the Coordinator receives assignment from management to lead the course, after which he/she:

  (1) Obtains commitment of the other members of the training team for the course, including Secretary, Instructors, and (if used) Laboratory Assistant;

  (2) Determines the composition and geographic origin of the student body to be trained;

  (3) Develops a working schedule and specific staff assignments for all significant milestone stages of course preparation and implementation;

  (4) Meets with course staff, distributes instructional guides and related training materials, and reviews the preparation plans and day-to-day working schedule; assigns specific topics and time allocation for which each Instructor has prime instructional responsibility and for which each Instructor serves as assistant to prime Instructor;

  (5) Identifies which, if any, available options in procedures should be taught for compliance with directives of the governing regulatory authority (-ies);

  (6) Prepares announcement of the course, and distributes it as appropriate to the potential student body;

  (7) Reviews classroom and laboratory facilities, making arrangements for any required repairs or adaptations;

  (8) In consultation with Instructors, reviews availability and condition of all equipment, supplies, and other training resources required for the course, and makes timely arrangements for repair and maintenance, reorder, or borrowing of needed items;

  (9) Arranges for supplies of student reference texts and associated training materials;

  (10) Works with Secretary in registration of students and in all pre-course communications on schedules, local housing and transportation, and other pertinent matters;

  (11) If the course is to be conducted in a "field" location, coordinates timely arrangements for staff travel, transportation of equipment and supplies, arrangements for training facilities, local housing and transportation, determination of availability and location of dining facilities, and any other preparations required for course conduct away from normal base location;

(12) Initiates periodic and timely checks with other training team members to assure that their pre-course preparations are on schedule and that preparation of required resources is moving ahead according to plan. Takes action as necessary to identify problems and to expedite solutions as the need arises;

(13) Represents the training team in all formal communications with management, host organizations, students, and with commercial or private sources of equipment and supplies.

b) During the course, the Course Coordinator:

(1) Takes charge of course opening exercises including student registration, welcome and staff introductions. Presents and discusses course objectives;

(2) Maintains general supervision of course, assuring that all activities are kept on schedule; maintains liaison between staff members and other interfacing organizations/individuals as necessary;

(3) Maintains consolidated summary record of student performance based on information provided by other Instructors; with staff determines which students should/should not be recorded as having satisfactorily met training objectives;

(4) Provides Course Secretary with timely information necessary for preparation of course certificates; signs course certificates as representative of the course training staff;

(5) Presides over course closing activities, including award of certificates.

c) After the course, the Course Coordinator:

(1) Reviews and evaluates with the instructional staff all matters considered pertinent to the effective implementation of the course as planned, developing proposals and plans for adjustments as necessary for future offerings of the course;

(2) Orders repair, renovation, and replacement of any equipment or supplies that require such attention;

(3) Coordinates return of any borrowed resources used in the course;

(4) If course was conducted in the field, coordinates repacking and return shipment of all equipment and supplies;

(5) Drafts course summary/evaluation report;

(6) Prepares and forwards any reports required by other supervising, coordinating, or financing authority.

2) Course Secretary

The Course Secretary works under the direct supervision of the Course Coordinator, and prepares or arranges preparation of all formal communications, procurement documents, training materials, and records required for course preparation and implementation. The Course Secretary also provides office support work for the course instructional staff in all matters pertaining to course preparation and implementation.

a) Before the course, the Course Secretary:

(1) Works with the Course Coordinator in identification and preparation of copies of all pertinent course materials for use in planning conferences between Course Coordinator and the instructional staff, including Instructors' copies of working schedules for course preparation, course agenda from preliminary to final draft, staff assignments, Instructors' instructional package worksheets, all student instructional materials and associated data sheets, student evaluation sheets, quizzes, and any other typed or printed material projected for course use;

(2) After Course Coordinator's conference with instructional staff, and resolution of decision making issues, arranges for printing (or reproduction) and assembly of all materials indicated under (1) above, in a quantity adequate for projected course requirements;

(3) Arranges for printing or reproduction, and distribution of the projected course announcement as directed by Course Coordinator;

(4) Serves as Registrar, maintaining roster and records of students submitting application and accepted for admission to the course, prepares routine response to students announcing acceptance with information as appropriate on course dates and schedule, local "geography" including key addresses, hotel/motel/dining information, local transportation information, and any other information which will simplify personal planning of registered students;

(5) Prepares, or arranges for, all individual student supplies, including registration cards, course manuals, note paper, pencils, name tags (1 for wearing and 1 for use at classroom seating position), course certificates, etc.;

(6) Prepares orders or procurement requests for equipment and supplies needed for the course based on specifications provided by the instructional staff;

(7) If the course is a "field" presentation, makes arrangements for shipment of equipment and supplies to course site and return, staff travel schedules and order of tickets, hotel reservations, and associated functions;

(8) Prepares the classroom for use in the course, including distribution of individual student materials to seating positions, arrangements for classroom organization of audiovisual projection or playback equipment, chalkboards and associated supplies, and other classroom needs. (Ordinarily, the Course Secretary does not travel to a "field" presentation; this function will have to be provided through special arrangements with the host organization at the course site.)

b) During the course, the Course Secretary functions as an "unofficial staff hostess," and:

(1) Attends course opening exercises, assisting students in completion of registration cards and associated records;

(2) Prepares course summary registration information, prepares class roster on first day of course and distributes copies to students and instructors, keeping copies for future records;

(3) Provides clerical/secretarial support to Course Coordinator and instructional staff as required;

(4) Receives incoming mail and messages for staff and students, expediting communications to extent practical and feasible;

(5) Prepares course certificates as specified by Course Coordinator on last day of course;

(6) Inspects classroom daily, making arrangements as necessary for coordination of deficiencies in janitorial services, and personally corrects minor deficiencies to extent feasible;

(7) Attends and participates in course closing exercises.

c) After the course, the Course Secretary:

(1) Prepares typed copy of all reports drafted by the Course Coordinator, and forwards reports as indicated;

(2) Places purchase orders or procurement requests for repair, renovation, or replacement of equipment and supplies as directed by Course Coordinator;

(3) Removes all course supplies from the classroom, returns borrowed resources, leaves classroom in condition suitable for use by following class (this does not imply janitorial services!);

(4) If course is conducted on a repetitive basis, inventories all consumable classroom supplies including data sheets, worksheets, quizzes, course schedules, and the like, and reorders or provides for reproduction of any items coming into short supply.

3) Instructors-Chemists

a) Before the course, each Instructor receives course duty assignment from management, and:

(1) Meets with Course Coordinator for discussions of course plans, objectives, and for development of day-to-day course preparation schedule;

(2) In conference with Course Coordinator determines which of any options in tests and measurements will be taught, determines lesson guides to be followed and student reference materials to be used, and resolves any other problems on instructional materials, their content, and related matters which should be determined beforehand;

(3) Reviews requirements for equipment, supplies, audiovisual training aids, and other training resources to be used in individual instructional assignments. Performs equipment upkeep and maintenance procedures, prepares supplies and reagents required to be available for student use. Provides Course Coordinator with timely, detailed information on specifications for all equipment, supplies and other training resources which must be purchased, rented, or borrowed for the course;

(4) If the course is to be conducted in a "field" location, packs equipment and supplies for shipment so that they will arrive at destination in good condition; identifies to the Course Coordinator the equipment and supplies which should not or cannot be shipped which should be provided otherwise at the course site;

(5) Rehearses all classroom and laboratory instructional presentations to the extent necessary to assure effective performance within the scheduled time allocation;

(6) Reviews and practices all tests for which he/she has responsibility as primary Instructor to assure personal proficiency and adequacy of pre-course plans and preparations. Supervises pre-course practice of those who will serve as assistant Instructors for the tests;

(7) Prepares to serve as assistant Instructor for specified tests and measurements, developing personal proficiency through pre-course practice under supervision of the applicable primary Instructor, and prepares to teach the tests and measurements in accordance with techniques specified by the primary Instructor;

(8) Reports periodically as requested to the Course Coordinator on status of course preparations, and cooperates in working out timely procedures for their accomplishment.

b) During the course, each Instructor:

(1) Attends and participates in course opening exercises;

(2) Serves as primary Instructor for the tests and measurements assigned to him/her. In this capacity he/she is responsible for all classroom instruction on the designated subjects, leadership of all laboratory instruction, collection of (or arrangements for) samples for laboratory examination by class, student performance evaluation and associated records for submission to Course Coordinator, and student counseling on pertinent matters related to area of personal responsibility;

(3) Assists primary Instructor in laboratory instruction phases of the tests and measurements as assigned, including preparation and organization;

(4) Attends and participates in all classroom start-of-day evaluation sessions on previous day's student performance;

(5) Attends and participates in course closing exercises.

c) After the course, each Instructor:

(1) Reviews the course implementation experience with the Course Coordinator, mutually developing proposals and plans for future offerings of the course;

(2) Evaluates condition of all equipment and supplies, initiating action to repair, renovate, or replace any items found deficient or in short supply;

(3) Takes necessary action to put laboratory into state of neatness and order for occupancy of the next course (this does not imply janitorial service!);

(4) If course was conducted in the field, repacks all equipment and supplies for return to home institution, after at least superficial cleaning of all dirty or contaminated glassware;

(5) On return of shipment to home institution, unpacks all equipment and supplies, returning it to designated custodial site, including return of borrowed equipment and other returnable resources.

4) Laboratory Assistant

The Laboratory Assistant is designated as "optional" in the staffing plan, but services of a Laboratory Assistant are strongly recommended. This is particularly urged in a fixed training installation where this and other courses are being conducted on a continuing or repetitive basis. The Laboratory Assistant works particularly in support of the instructional staff. The Assistant will be given routine tasks which will free the instructional staff for more specialized or complicated tasks associated with the planning, preparation, and implementation of the training.

a) Before the course, the Laboratory Assistant:

   (1) Works closely with the instructional staff members, performing standardized tasks as specified in the course preparation plan;

   (2) Organizes laboratory supplies and equipment for each procedure in such a way as to permit distribution to the students or to their working sites with maximum efficiency during the course presentation;

   (3) Assists Course Secretary wherever feasible in assembly and organization of student instructional materials, classroom preparation, and related tasks.

b) During the course, the Laboratory Assistant:

   (1) Performs all possible tasks in support of primary Instructors in setting up student work positions, collecting and returning used glassware, supplies and equipment, etc., to central repository;

   (2) Cleans and maintains all glassware and supplies excepting those stipulated for student performance;

   (3) Notifies primary Instructor promptly of any noted discrepancies or deficiencies in supplies, equipment, or planning which would lead to problems in implementing the course;

   (4) In field courses, packs equipment in shipping cases as rapidly as its use has been completed for the course.

c) After the course, the Laboratory Assistant:

   (1) Assists Instructors in all equipment and supply inspection, renovation, and return to proper location;

   (2) Puts laboratory in state of neatness and order preparatory to use for next class;

   (3) Prepares any stable supplies required for next offering of the course, within limits of technical capability.

2. Training Facilities

This course requires both a classroom and a laboratory for class use. Effective presentation of the course requires staff attention to many details related to these facilities. Problems more often occur in field courses (i.e., away from "home base"). In any location it is unusual that all desired features of a training facility will be met, but with timely attention most problems can be solved or at least partially resolved.

a. General Considerations

1) Spatial Relationships

Classroom and laboratory should be separate, but close together. Much of the instruction requires frequent shifts between classroom and laboratory. Therefore, the classroom and laboratory must not be in separate buildings, and should not be far apart within any structure.

2) Associated Comforts

a) The classroom and the laboratory should have a comfortable temperature, be free of obvious drafts, be well-ventilated, and should be well-lighted. It is, of course, possible to develop specifications for acceptable temperature ranges, light intensity ranges, humidity, etc.; but there is no substitute for exercise of good judgment.

b) Suitable restroom and drinking fountain facilities should be convenient to the classroom and laboratory.

c) Smoking:

(1) NO SMOKING IN THE LABORATORY. There should be no compromise on this.

(2) Some schools permit smoking in the classrooms. If this is the practice, it is advisable to locate ashtrays so that smokers sit in an area where their smoking will not disturb others.

3) Lunchroom Facilities

Most schedules for this course will allow a one-hour lunch break. It is advisable that the course staff identify and make known to the class the names and locations of convenient dining facilities where service, variety, quality, and price are satisfactory.

4) Comments to Class about Facilities

a) On the first day of the course the general orientation should include such information as the class needs on the location and use of facilities and conveniences for class use.

b) It is strongly urged that the entire strining staff never at any time indulge in apologies or criticisms of the classroom or laboratory facilities being used. Such remarks serve no useful prupose and can only detract from an effective program, provided that everything possible has been done beforehand to resolve existing problems with facilities. Student comments and complaints should be given an honest repsonse, but such comments from students should not be regarded as an excuse for staff to enlarge on the subject.

b. Classroom

1) General Features

a) Door at rear of room is preferred; this permits entry of latecomers without excessive distraction of class.

b) The classroom should be free from excessive extraneous noises, such as from construction projects, heavy traffic, or from aircraft.

c) The classroom should have adequate electric power outlets (115 V) for use of audiovisual equipment. The receptacles should be inspected for assurance that they are compatible with the plugs on the audiovisual projector equipment being used, and adapters and extension cords secured as required.

d) Room size should be adequate for seating 18 students, plus providing for instructor equipment, projection equipment, and a modest number (4 to 8) of intermittent visitors to the classroom.

e) The classroom should be capable of being darkened quickly and effectively for use of projection equipment or television. Room dimmer lights for indirect lighting (not striking the screen directly) are recommended in fixed training installations, but can be dispensed within a field training situation.

2) Student Facilities

a) Ideally, students should be seated at tables, with all seats facing the instructor's area at the front of the classroom. Each student should be allocated 30" or more of table width. The sidearm chairs so familiar in the classrooms of secondary schools and colleges may be used if absolutely necessary, but are distinctly inferior to tables for student work.

b) Student seating should be at least two screen widths from the projection screen (assuming a 6' screen, no student closer than 12' from the screen) and not more than 6 screen widths from the screen (again assuming a 6' screen, no student more than 36' from the screen). Furthermore, all students should be seated seated within a 30° angle to the left and to the right of a line from the middle of the projection screen to the projector.

3) Classroom Instructional Facilities

a) Lectern, either freestanding or table-type; suitable for standing instructor

b) Demonstration table at front of classroom, approximately 3' x 5'

c) Chalkboard, at least 3' x 5' (preferably larger), with chalk, erasers, pointer

d) Audiovisual equipment

(1) Public address system (optional but recommended) with lavalier microphone with adequate cord length to permit instructor to move about at front of classroom with relative freedom

(2) Projection screen (for size consideration see 2) b above), matte, beaded, or lenticular surface

(3) Projector, 35-mm slide projector for slides mounted in cardboard or plastic mount; carousel type preferred. Should have projection lens with cord length suitable for use from rear of room

(4) Projector, overhead type, for use with projectuals approximately 7" x 9"

(5) Cassette type playback unit, with cueing feature for automatic operation of cassette type slide projector; compatible with National Training and Operational Technology Center (EPA) tape/slide instructional units

(6) Television tape playback unit (3/4" cassette type, "U-Matic" or equivalent)

(7) Television receiver, commercial type, color, 19" diagonal picture, or larger At least one receiver, preferably two

c. Laboratory

1) General Features

a) Should be well-lighted, adequately ventilated. It is particularly important that the laboratory be free from strong drafts in student working areas.

b) Should provide for students to stan [illegible] aboratory benches which are approximately 36" from floor to [illegible] surface.

c) Conventional laboratory services should be available at student work areas, including electricity (115 V), gas, and vacuum.

d) Space between benches should be adequate for students to work without interfering with each other, and to permit free movement of instructors in the student working area.

e) Safety features of the laboratory should be checked, including location and condition of first aid kits, fire extinguishers, emergency showers, eye-wash facilities, and other emergency equipment.

2) Student Facilities

a) Provide at least 6' of bench width per student pair. While students will work in pairs to the extent that they will share certain limited equipment, each student will perform all tests and measurements.

b) Provide bench space or floor space as necessary for laboratory equipment described in the equipment and supply lists, such as balances, ovens, waterbaths and other items not assigned to individual student work.

3) Laboratory Instructional Facilities

a) A chalkboard and demonstration table are recommended.

b) Provide at least 20 square feet for reserve supplies and equipment of each instructor.

c) For field courses, provide area for packing and unpacking; areas for equipment to be shipped. This should be at least 100 square feet of floor space, with at least 20 square feet of table space.

d. Security

Valuable property is used both in the classroom and in the laboratory. Some of the items are particularly susceptible to theft. Accordingly:

1) Provide for locking of both classroom and laboratory when not in use, or assure that adequate security is provided in the facility by other means.

2) Be sure that the necessary keys are available to the instructional staff at their need.

3) With field courses, often it is necessary for the training staff to work in the evening or weekends to prepare for coming classwork. Arrangements must be made well in advance to secure authorized entry to the training facilities being made available by a host organization.

4) Thefts during normal working hours may be a special problem. Maintain surveillance to the extent practical, and keep out-of-service theft-prone items out of exposed locations.

3. Laboratory Equipment and Supply Requirements

The consolidated list in this section is for overall planning purposes. For day-to-day laboratory requirements, see the "IPW Equipment and Supply Requirements" and the "IPW Reagent Requirements" in each Instructional Package Worksheet contained in Part II of this Guide.

Before using the listing in this section decisions must be made about the specific procedure to be taught for each test in the course because several of the Effluent Monitoring Procedures (EMPs) contain a choice of procedures. The Ammonia EMP is written for the distillation of a macro sample followed by either Nesslerization or titration. The Kjeldahl Nitrogen EMP contains a choice between macro or micro digestion and distillation, followed by either Nesslerization or titration. There is an EMP for determining ammonia by using a selective ion electrode. The Chemical Oxygen Demand EMP is written for the low level range of 5-50 mg/liter but includes the modifications for concentrations greater than 50 mg/liter COD. The Phosphorus EMP can be used to determine total phosphorus or only orthophosphate. These different possibilities affect the equipment and supply needs. The requirements of area NPDES permits and/or the type of equipment readily available will help the course planners make the choices among these possibilities.

The quantities of laboratory materials needed for a course depend on the specific laboratory work assigned to each student. For the consolidated list in this section, the basis for quantities required is the assignment stated in each Instructional Package Worksheet (IPW).

The abbreviations used in the listing to refer to the various procedures that might be taught and the assignment given for each in the related IPW are stated in a table preceding the laboratory listing.

The listing itself is divided into three sections: capital equipment of more than $100 unit value, reusable equipment of less than $100 unit value, and consumable equipment of less than $100 unit value. The first column in each section contains a description of the item in the language and the qualifications required for ordering the item from commercial catalogs. The second column lists the minimum quantity per student required for each procedure presented in the EMPs for the course. The third column lists the minimum quantity for a class of 18 students. The numbers found here are often based on multiple use of the item. The fourth and final column contains remarks that may be useful when deciding on class needs or when ordering equipment.

As noted, numbers represent minimum quantities. It is strongly recommended that instructors provide surplus equipment and additional supplies ready for use in case of need. Many instructors plan for a margin of at least 10% of extra supplies to provide for student errors, planning miscalculations, or other unforeseen events.

This list can be of great value in pre-course planning, to determine the availability of needed equipment and supplies, and to take action to provide needed resources. Further, this list can be of vital importance when planning for courses to be conducted in field locations. Copies of the list in the hands of the Course Coordinator and a representative of the host organization can be used to determine which will provide needed resources, on an item-by-item basis. When the responsibility is assigned/accepted, this can be annotated in the "remarks" column, with a copy of the annotated list in the hands of the Course Coordinator and a copy for the representative of the host organization. Each can then use the annotated equipment and supply list as a checklist for carrying out his own agreed-upon responsibilities in preparing for the course.

LABORATORY EQUIPMENT AND SUPPLY REQUIREMENTS

Following is the key for abbreviations used for course topics. Quantities per test are based on the IPW assignments as noted.

ABBREVIATIONS FOR TESTS		IPW ASSIGNMENTS
ADma	Ammonia Distillation, macro	1 sample per 2 students
ADmi	Ammonia Distillation, micro (as part of Kjeldahl N EMP)	1 sample
AE	Ammonia Electrode	2 standards + 2 samples
AN	Ammonia Nesslerization (as part of Kjeldahl N EMP)	1 blank + 2 standards + 1 digested sample
ATma	Ammonia Titration, macro	1 sample
CG	Calibration Graphs	1 graph
COD	Chemical Oxygen Demand (low level)	1 sample or blank 1 FAS standardization
KDma	Kjeldahl Digestion, macro (alternative to micro)	1 sample
KDmi	Kjeldahl Digestion, micro	1 sample
NO_3	Nitrate + Nitrite and Nitrate	Prepare one reduction column. 2 blanks + 2 standards 2 samples
OG	Oil and Grease	1 sample
OP	Orthophosphate as P (alternative to Total Phosphorus)	1 blank + 1 standard 1 sample
SP	Spectrophotometer	1 instrument
TP	Total Phosphorus as P	1 blank + 1 standard 1 sample

A. CAPITAL EQUIPMENT (More than $100 Unit Value per One Item)

	QUANTITY FOR:		
DESCRIPTION	EACH STUDENT	CLASS OF 18	REMARKS
BALANCES, analytical, 0.1 mg sensitivity at a load of 200 g, with Instruction Manuals	OG 1/4 OP 1/3 TP 1/3	6	
BALANCES, Trip, 500 g capacity	NO_3 1/3	6	To weigh cadmium
DIGESTION APPARATUS, for Kjeldahl determination. See diagrams of equipment in EMP. Quantity is according to heat sources required.	KDma 1 KDmi 1	18	Apparatus is readily available commercially.
DISTILLATION APPARATUS, for ammonia and/or Kjeldahl determinations. See diagrams of equipment in Kjeldahl Nitrogen EMP.	ADmi 1/4	4	ADmi set-ups shown in EMP require some fabricated parts. (Inquire EPA-NTOTC) ADmi total assemblies are also available commercially.
ELECTRICAL OUTLETS, 120 volts	TP 1	?	Number depends on number (size) of HOT PLATES used.
HEATING SURFACES, flat for Erlenmeyer flasks or else heating mantles for round bottom flasks. Choice should produce at least 9 watts/sq. inch and have adjustable heat control.	COD 1	18	A 16 amp line is usually required for a series of 6 reflux set-ups.
HOT PLATES with continuous settings, not just "low," "medium" and "high." If doing AE EMP, magnetic stirring feature is desirable.	OG 1 TP 1	?	Number depends on size. OG for 1 - 1 liter beaker for each, TP for 3 - 125 ml Erlenmeyers for each.
HOOD SPACE or equivalent well-ventilated area.	OG 1 OP 1 - 1/6 TP 1 - 1/6	1-3	OP/TP: three are needed if students clean glassware.

	QUANTITY FOR:		
DESCRIPTION	EACH STUDENT	CLASS OF 18	REMARKS
pH METERS, ELECTROMETRIC accurate to at least 0.1 pH unit and preferably with a single electrode	NO_3 1 OP 1 TP 1	18	To conserve time, this quantity is preferable.
SPECIFIC ION METERS, Orion* Model 401, 407 or 407A, each with AMMONIA ELECTRODE, Orion* Model 95-10	AE 1/3	6	Sufficient if alternating use with other lab work
SPECTROPHOTOMETERS, Bausch and Lomb* Spectronic 20 or equivalent, with Instruction Manuals	AN 1/3 NO_3 1/3 OP 1/3 SP 1/3 TP 1/3	6	
SPECTROPHOTOMETER CELLS for instruments used	AN 1/3 NO_3 1/3 OP 1/3 SP 1/3 TP 1/3	6	If matched sets of cells are available, apply quantities to sets.
SPECTROPHOTOMETER PHOTOTUBES, Infrared range (B&L* #33-29-72) plus infrared FILTERS (B&L* #33-29-18) or equivalent	OP 1/3 SP 1/3 TP 1/3	6	
SPECTROPHOTOMETER PHOTOTUBES, visible range (B&L* #33-29-71) or equivalent	AN 1/3 NO_3 1/3 SP 1/3	6	
VACUUM SOURCES: preferably a central service. An electric vacuum pump assembly with suitable hoses, water traps and shut off valves and with capability of drawing 15 inches of mercury can be used.	OP 1/3 TP 1/3	6	OP for filtration of turbid samples
WATER BATHS to maintain 80°C for 125 ml distilling flasks	OG 1	?	Number depends on size: 1 - 125 ml flask for each student
WATER STILL with an anion-cation exchange system to produce water free of ammonia	ADma 1 ADmi 1 AE 1 AN 1 KDma 1 KDmi 1 ATma 1	1	

*Mention of a specific brand name does not constitute endorsement of the U.S. Environmental Protection Agency

B. REUSABLE EQUIPMENT (Less than $100 Unit Value)

DESCRIPTION	QUANTITY FOR: EACH STUDENT	CLASS OF 18	REMARKS
APRONS, laboratory (plastic acceptable)	All 1	18	
BEAKERS, glass, 50 ml	ADma 1 ADmi 1	18	
BEAKERS, glass, 150 ml	AE 4 COD 1 NO_3 1 OG 2	36-72	72 only if AE is taught
BEAKERS, glass, 250 ml	NO_3 3	54	
BEAKERS, glass, 600 ml	ADma 1/2 NO_3 1 OP 1 TP 1	18	OP/TP only if cleaning glassware
BEAKERS, glass, 1000 ml	OG 1	18	
BOILING BEADS, glass 5 mm	ADma 6 ADmi 6 COD 5 KDma 6 KDmi 6 TP 12	738	
BOTTLES, glass dispensing, 100 ml	AT 1/2	9	
BOTTLES, glass, for aliquots of samples, 225 ml minimum	COD 1/3	6	Each needs 75 ml
BOTTLES, plastic, for aliquots of samples, 250 ml minimum	KDmi 1/3 NO_3 1/3 OP 1/3 TP 1/3	18	Each test volume is 50 ml per one student.
BOTTLES, plastic, for aliquots of samples, 1000 ml	KDma 1/2 ADma 1/2 AE 1/3	24	Macro tests require 400-500 ml. AE requires 2 - 100 ml samples for each.
BRUSHES, assorted, for cleaning glassware	All 1/2	9	

DESCRIPTION	QUANTITY FOR: EACH STUDENT	CLASS OF 18	REMARKS
BRUSHES, balance	NO_3 1 OG 1 OP 1 TP 1	12	Put by balances.
BURETS, 25 ml, 0.1 ml graduations, teflon stopcock plug preferred	COD 1	18	
BURETS, 50 ml, 0.1 ml graduations, teflon stopcock plug preferred	ATma 1	18	
BURNERS, gas, Meker type or equivalent heat source	ADma 1/2 ADmi 1/6	9	
CLAMPS, buret for titration stand, to hold two burets	ATma 1/2 COD 1/2 NO_3 1/2	9	
CLAMPS, for titration stand to fit neck of 125 ml distilling flask	OG 1/2	9	
CLAMPS, screw to regulate flow through 4 cm ID rubber tubing	NO_3 1	18	
CLAMP, pinchcock for 4 cm ID rubber tubing	NO_3 1	18	
CONDENSERS, 12 inch Allihn or equivalent with a ground glass joint to fit into heat-resistant FLASK. (24/40 is a commonly used joint size)	COD 1	18	
CONDENSERS, 16 inch Graham or Allihn. Extend tip with rubber and glass tubing for delivery into 500 ml graduated Erlenmeyer FLASK.	ADma 1/2	9	
CONTAINERS for acid and mercury wastes, about 2 liter	COD 1/4	5	Up to 400-500 ml per student

	QUANTITY FOR:		
DESCRIPTION	EACH STUDENT	CLASS OF 18	REMARKS
CYLINDERS, graduated, 10 ml	ADmi 2 KDmi 1 OG 1	36	
CYLINDERS, graduated, 25 ml	COD 1	18	
CYLINDERS, graduated, 50 ml	NO_3 1 OG 1	18	
CYLINDERS, graduated, 100 ml	ADma 2 AE 1 COD 1 KDma 1 KDmi 1 NO_3 1 OG 1	18-36	36 only if ADma is taught
CYLINDERS, graduated, 500 ml	ADma 1 KDma 1	18	
DESICCATORS with effective desiccant and large enough for 1 - 125 ml distilling flask per student	OG 1	?	Number depends on size of desiccator
EVAPORATING DISHES, or similar pieces to place between heating surface and flask for cooling	COD 1	18	
FILTERING ASSEMBLIES for 60 ml or 10 ml volumes, filtrate to be transferred	OP 1 TP 1	18	
FLASKS, distilling, 125 ml with a 24/40 ground glass neck (Corning* #4100 is an example)	OG 1	18	
FLASKS, Erlenmeyer, wide mouth, 125 ml	OP 3 TP 3	54	
FLASKS, Erlenmeyer, wide mouth, 250 ml	NO_3 3	54	

*Mention of a specific brand name does not constitute endorsement of the U.S. Environmental Protection Agency.

DESCRIPTION	QUANTITY FOR: EACH STUDENT	CLASS OF 18	REMARKS
FLASKS, Erlenmeyer, wide mouth, graduated, 500 ml	ADma 1 COD 1	18	
FLASKS, heat resistant glass, 500 ml graduated Erlenmeyer or 300 ml round bottom with a ground glass neck to fit CONDENSER of choice.	COD 1	18	
FLASKS, Kjeldahl, 100 ml	ADmi 1 KDmi 1	18	
FLASKS, Kjeldahl, 800 ml	ADma 1 KDma 1	18	
FLASKS, volumetric, 50 ml, with glass stoppers	NO_3 1 OP 3 TP 3	54	
FLASKS, volumetric, 100 ml with glass stoppers	NO_3 2	36	
FLASKS, volumetric, 1000 ml with glass stoppers	OP 1 TP 1	18	If student does balance and/or dilution exercises
FORCEPS, pairs to manipulate filter paper	OP 1 TP 1	18	
FUNNELS, Buchner, to fit CONTAINER for COD wastes (for glass beads)	COD 1/4	5	
FUNNELS, separatory, 2 liter with Teflon stopcock	OG 1	18	
FUNNELS, short stem, diameter about 50 mm to fit neck of 50 ml volumetric flask and 25 or 50 ml buret	COD 1 OG 1 OP 1 TP 1	18	
GLASSES, pairs of safety	All 1	18	

DESCRIPTION	QUANTITY FOR: EACH STUDENT	QUANTITY FOR: CLASS OF 18	REMARKS
GLOVES, pairs of rubber material	NO_3 1 TP 1 OP 1	18	
MAGNETIC STIRRERS with magnetic bars	AE 1/3	6	Note HOT PLATES in A, Capital Equipment
NESSLER TUBES, scored at 50 ml, low form desirable	AN 3 KDmi 1	72	
NESSLER TUBE SUPPORTS	AN 1/2	9	
PANS for ice water to cool 500 ml Erlenmeyer flasks	COD 1/2	9	
PIPETS, dropping (medicine droppers) with bulb about 1 ml	OG 1	18	
PIPETS, measuring, Mohr glass, 1 or 2 ml, graduated at 0.5 ml	NO_3 1/4	5	
PIPETS, measuring, glass, 1 ml graduated at 0.1 ml	OP 1 TP 1	18	To adjust pH
PIPETS, measuring, Mohr glass, 10 ml	AN 2 OP 3 COD 1 TP 3 NO_3 1	54	
PIPETS, volumetric transfer, 1 ml	AE 1 OP 1/8 NO_3 1/4 TP 1/8	5-18	18 only if AE taught
PIPETS, volumetric transfer, 2 ml	NO_3 1/4	5	
PIPETS, volumetric transfer, 3 ml	OP 1/8 TP 1/8	3	

	QUANTITY FOR:		
DESCRIPTION	EACH STUDENT	CLASS OF 18	REMARKS
PIPETS, volumetric transfer, 5 ml	NO_3 1/4 OP 1/8 TP 1/8	5	
PIPETS, volumetric transfer, 10 ml	COD 1 NO_3 1/4 OP 1/8 TP 1/8	18	
PIPETS, volumetric transfer, 20 ml	OP 1/8 TP 1/8 OP 1 TP 1	3-18	18 only if students dilute stock solution
PIPETS, volumetric transfer, 25 ml	COD 1 NO_3 1	18	
PIPETS, volumetric transfer, 30 ml	OP 1/8 TP 1/8	3	
PIPETS, volumetric transfer, 40 ml	OP 1/8 TP 1/8	3	
PIPETS, volumetric transfer, 50 ml	COD 1 NO_3 3 OP 1 TP 1 OP 1/8 TP 1/8 KD mi 1	54	
PIPETS, volumetric transfer, 100 ml with top and tip cut off for reduction column	NO_3 1	18	
PIPETS, volumetric transfer of size required if sample dilution is necessary	KD mi 1 OP 1 TP 1	18	
PIPET BULBS, large with shaped opening to fit variety of pipet sizes	All 1	18	
PROPIPET BULB	NO_3 1	18	
PIPETTERS, automatic (OPTIONAL), glass with delivery setting for at least 10 ml, for acids	COD 2/6	6	

DESCRIPTION	QUANTITY FOR: EACH STUDENT	CLASS OF 18	REMARKS
REAGENT SPOONS, 0.4 gram	TP 1/3	6	
REAGENT SPOONS, 1 gram	COD 1/3	6	
RINGS, metal for stand to support a 2 liter separatory funnel	OG 1	18	
RULERS, 12 inches long	CG 1	18	
SIEVES, 60 mesh	NO_3 1	18	
SPATULAS	NO_3 1 Balances 12	30	
SPRAY TRAP, Kjeldahl, connecting bulb, 55 mm. See diagram in Kjeldahl Emp.	ADma 1/2	9	
STANDS, titration with porcelain bases	ATma 1/2 COD 1/2 NO_3 1/2 OG 1	18	
STEAM BATHS for 125 ml distilling flask	OG 1	?	Number depends on size of bath
STOPPER/GLASS TUBING APPARATUS for applying suction to a 125 ml distilling FLASK. See OG EMP, Figure 2.	OG 1/6	3	
STOPPERS, rubber #3 or #6 to fit NESSLER TUBES	AN 1	18	
STOPPERS, rubber #3 to fit CONDENSER (Allin) for spray trap connection	ADma 1/2	9	
STOPPERS, rubber #7 to fit neck of 800 ml Kjeldahl FLASK for spray trap connection	ADma 1/2	9	
TONGS, crucible, pairs	TP 1	18	

DESCRIPTION	QUANTITY FOR: EACH STUDENT	CLASS OF 18	REMARKS
TUBING, soft rubber, 4 cm ID, 7.5 cm length	NO_3 1	18	
TUBING, tygon for CONDENSER as connection to cooling water source. Lengths depend on apparatus	COD 1	18	
WASH BOTTLES, plastic squeeze type, 500 ml	All 1	18	

C. CONSUMABLE SUPPLIES (Less than $100 Unit Value)

FILTER DISCS, Whatman No. 40 to fit FUNNEL with diameter of about 50 mm	OG 1	18	
FILTER DISCS, phosphorus-free, 0.45 micron pore size, Gelman GA-6 or equivalent, to fit FILTERING ASSEMBLIES	OP 1 TP 3	18-54	54 for TP
GLASS WOOL, small wad for end of 100 ml cut-off pipet	NO_3 1	18	
GRAPH PAPER, sheets, arithmetic	CG 1	18	Can substitute printed graph sheets in EMP
ICE SUPPLY, trays of cubes	COD 1/3	6	
PENCILS, WAX MARKING	All 1	18	
pH PAPER, strips, short range to test at pH 9.5	ADma 5	90	
REAGENTS are listed according to test at the end of this section			
SAMPLES are listed according to test at the end of this section			

DESCRIPTION	QUANTITY FOR: EACH STUDENT	CLASS OF 18	REMARKS
SAMPLE TAGS, preferably pre-printed with blanks for information required by NPDES reports	All	~45	
TISSUES, soft in boxes	SP 6 BALANCES 6	12	
TOWELS, paper, in boxes or packets	SINKS 1	3	
WATER, distilled, ammonia-free	ADma 0.1 l ADmi 0.1 l AE 0.5 l AN 0.15 l	13.5 l	
WATER, distilled, ammonia-free and nitrate-nitrite free	NO_3 4 l	72 l	The large quantity is required to prepare Cu-Cd.
WATER, distilled, phosphorus-free	OP 2.5 l TP 2.5 l	45 l	The large quantity is required for cleaning glassware.
WATER, distilled, negligible organic concentrations	COD 0.2 l	~4 l	
WATER, distilled	SP 0.1 l	~2 l	
WEIGHING BOATS, disposable	NO_3 1	18	To weigh cadmium

REAGENTS:

AMMONIA DISTILLATION, macro or micro (AD is used to isolate ammonia from samples. It is also used to isolate ammonia from digested samples as part of the Kjeldahl N test.)	Amounts are minimum		See p. 160 or p. 176 EPA Methods Manual. Also see Ammonia EMP or the Kjeldahl N EMP, as applicable.
Boric Acid solution, 2%: 10 g boric acid/500 ml	ADma 50 ml ADmi 5 ml	450 ml 90 ml	For ADmi, make 250 ml.

	QUANTITY FOR:		
DESCRIPTION	EACH STUDENT	CLASS OF 18	REMARKS
1N sodium hydroxide solution: 10 g sodium hydroxide/250 ml	ADma ~10 ml	~180 ml	For pH adjustment
Borate buffer solution - 500 ml: ~0.5 g sodium hydroxide ~2.5 g sodium tetraborate	ADma 25 ml	225 ml	For ADma, it is convenient to make the 500 ml volume.
Sodium hydroxide-sodium thiosulfate solution - 250 ml: 125 g sodium hydroxide ~7 g sodium thiosulfate pentahydrate	ADma 100 ml ADmi 10 ml	1800 ml 180 ml	For AD as part of Kjeldahl-N test. See that EMP.
Phenolphthalein solution: 1 g phenolphthalein/100 ml	ADma 10 drops ADmi 2 drops	180 drops 36 drops	For AD as part of Kjeldahl-N test. See that EMP.
AMMONIA ELECTRODE	Amounts are minimum		Preparations are according to electrode manufacturer. See EMP.
Stock ammonium chloride solution: 0.3819 g ammonium chloride/100 ml	10 ml	10 ml	Dilute this twice for 200 ml of 1.0 and of 0.1 mg/liter for each student for standards plus sample preparations.
10 M sodium hydroxide solution: 80 g sodium hydroxide/200 ml	4 ml	72 ml	
AMMONIA NESSLERIZATION	Amounts are minimum		See p. 177 in 1974 EPA Methods Manual. Also see Kjeldahl-N EMP.
Nessler reagent - 100 ml: 10 g mercuric iodide 7 g potassium iodide 16 g sodium hydroxide	4 ml	72 ml	
Stock ammonium chloride: 0.3819 g ammonium chloride/100 ml	0.18 ml	3.25 ml	Dilute this for up to 18 ml of 0.01 mg/1 per student.

DESCRIPTION	QUANTITY FOR:		REMARKS
	EACH STUDENT	CLASS OF 18	
AMMONIA TITRATION, macro	Amounts are minimum		See p. 160 in 1974 EPA Methods Manual. Also see Ammonia EMP.
Mixed indicator solution - 150 ml: 200 mg methyl red 100 mg methylene blue	3 drops	27 drops	
0.1 N sulfuric acid solution: 1.5 ml concentrated sulfuric acid/500 ml	To make 1 ml of 0.02 N per 0.28 mg/l NH_3-N	810 ml (max.)	Dilute this for up to 90 ml of 0.02 N acid per student.
CHEMICAL OXYGEN DEMAND, low level	Amounts are minimum		See p. 22 in 1974 EPA Methods Manual. Also see EMP.
Mercuric sulfate	1 g	18 g	
Concentrated sulfuric acid	25 ml	450 ml	OPTIONAL: Place in automatic pipetter and check for 5 ml delivery.
Sulfuric acid - silver sulfate solution - 2 liters: 2 liters concentrated sulfuric acid 21.6 g silver sulfate	70 ml	1260 ml	OPTIONAL: Place in 2 automatic pipetters and check for 10 ml delivery.
0.250 N potassium dichromate solution 2.452 g potassium dichromate/200 ml	100 ml	200 ml	Dilute this for 50 ml of 0.025 N per student.
Ferroin indicator solution - 100 ml: 1.48 g 1-10 orthophenanthroline monohydrate 0.70 g ferrous sulfate heptahydrate	11 drops	198 drops	Ferroin solution can be purchased.
0.250 N ferrous ammonium sulfate solution - 200 ml: 19.6 g ferrous ammonium sulfate hexahydrate 4 ml concentrated sulfuric acid	100 ml	200 ml	Dilute this for 75 ml of 0.025 N per student

DESCRIPTION	QUANTITY FOR: EACH STUDENT	 CLASS OF 18	REMARKS
KJELDAHL DIGESTION	Amounts are minimum		See p. 176 in 1974 EPA Methods Manual. Also see EMP.
Digestion reagent - 2000 ml: 267 g potassium sulfate 400 ml concentrated sulfuric acid 4 g mercuric oxide 5 ml concentrated sulfuric acid for 20% solution	KDma 100 ml KDmi 10 ml	1800 ml 180 ml	For KDmi, use 0.1 amounts to make 200 ml.
NITRATE + NITRITE and NITRATE	Amounts are minimum		See p. 204 in 1974 EPA Methods Manual. Also see EMP.
Concentrated ammonium chloride - EDTA solution - 10 liters 130 g ammonium chloride 17 g disodium ethylenediamine tetraacetate $\sim$10 ml concentrated ammonium hydroxide	480 ml	8640	Dilute this for 800 ml dilute solution per student.
Color reagent - 500 ml: 50 ml concentrated phosphoric acid 5 g sulfanilamide 0.5 g N-(1-napthyl)-ethylenediamine dihydrochloride	12 ml	216 ml	
Concentrated ammonium hydroxide	$\sim$10 ml	$\sim$180 ml	For pH adjustment
Concentrated hydrochloric acid	$\sim$10 ml	$\sim$180 ml	For pH adjustment
6 N hydrochloric acid 600 ml concentrated hydrochloric acid/1200 ml	60 ml	1080 ml	
2% copper sulfate solution: 80 g copper sulfate/4 l	200 ml	3600 ml	
Stock potassium nitrate solution: 3.609 g potassium nitrate/500 ml (1 ml chloroform as preservative)	11 ml	11 ml	Dilute this for 25 ml of 1 mg/l and for up to 15 ml of 0.01 mg/l and for 50 ml sample per student.

DESCRIPTION	QUANTITY FOR:		REMARKS
	EACH STUDENT	CLASS OF 18	
Stock potassium nitrite solution: 3.036 g potassium nitrite/500 ml (1 ml chloroform as preservative)	10 ml	10 ml	Dilute this for up to 15 ml of 0.01 mg/1 and for 50 ml sample per student
Cadmium,coarse powder, 40-60 mesh can be filed off stick. Should pass a 10 mesh sieve and be retained on a 40, then a 60 mesh sieve.	20 g	360 g	Powder is available from EM Laboratories*, 500 Executive Blvd., Elmsford, NY 10523.
pH buffers 4, 7 and 10	~100 ml of each	~1500 ml of each	For students to calibrate pH meters
OIL AND GREASE	Amounts are minimum		See p. 230 in 1974 EPA Methods Manual. Also see EMP.
1,1,2-trichloro-1,2,2-trifluoroethane Du Pont*, Freon 113 TF or Allied Chemical*, Genosolv D	125 ml	2250 ml	
Anhydrous sodium sulfate	3 g	54 g	
SPECTROPHOTOMETER	Amounts are minimum		See IPW for preparation
Set of synthetic phosphorus standards made from a methylene blue indicator which is diluted to give a series of standards for each instrument.	~1 g	~1 g	This is enough for several sets of "standards."
TOTAL PHOSPHORUS or ORTHOPHOSPHATE	Amounts are minimum		See p. 252 in 1974 EPA Methods Manual. Also see EMP.
1:1 hydrochloric acid (to clean glassware): 1500 ml concentrated hydrochloric acid/3000 ml	150 ml	2700 ml	
10 N sodium hydroxide solution: 40 g sodium hydroxide/100 ml	5 ml	100 ml	Use 10 ml for 5 ml of 0.1 N per student.

*Mention of a specific brand name does not constitute endorsement of the U.S. Environmental Protection Agency.

DESCRIPTION	QUANTITY FOR:		REMARKS
	EACH STUDENT	CLASS OF 18	
11 N sulfuric acid: 31 ml concentrated sulfuric acid/100 ml	4 ml	82 ml	Use 10 ml for 5 ml of 1.1 N per student.
Combined reagent - 3 liters: 210 ml concentrated sulfuric acid 0.6857 g antimony potassium tartrate 20 g ammonium molybdate 1.76 g ascorbic acid	150 ml	2700 ml	Includes 125 ml per student to check glassware.
21.6 g ammonium persulfate	1.2 g	21.6 g	
Stock phosphorus solution: 0.2197 g potassium dihydrogen phosphate/l	20 ml 30 ml	40 ml 540 ml	Dilute this for up to 50 ml of 1.0 µg /l per student. Make 30 ml per student if they dilute the stock.
Potassium dihydrogen phosphate	0.2197 g	∿4 g	For analytical balance exercise. Students can make own stock solution with this.
SAMPLES	Amounts are minimum		Distribute in plastic BOTTLES except the COD sample.
Sample, Ammonia Distillation, macro, followed by Ammonia Titration: WWT plant effluent with at least 1 mg/l NH_3-N	400 ml	∿9 l	Test range is 1 to 25 mg/liter ammonia nitrogen
Sample, Ammonia Electrode:			Dilute the standard ammonium chloride solutions.
Synthetic, between 0.03 to 0.1 mg/l NH_3-N	100 ml	∿2.5 l	
Synthetic, between 0.1 to 1.0 mg/l NH_3-N	100 ml	∿2.5 l	
Sample, Chemical Oxygen Demand: WWT plant effluent of 5-50 mg/l COD	75 ml	∿2 l	Distribute in glass BOTTLES.

	QUANTITY FOR:		
DESCRIPTION	EACH STUDENT	CLASS OF 18	REMARKS
Sample, Kjeldahl Digestion, micro, followed by Ammonia Distillation, micro, and Ammonia Nesslerization: WWT plant effluent of 0.03-1.0 mg/l NH_3-N	50 ml	$\sim$1.5 l	
Sample, Nitrate + Nitrite and Nitrate: Synthetic sample of up to 1 mg/liter $NO_2 + NO_3$ Example: 40 ml NO_3 standard plus 40 ml NO_2 standard into 1 liter gives 0.8 mg/l total.	50 ml	$\sim$1.5 l	Dilute the standard potassium nitrate and potassium nitrite solutions.
Sample, Oil and Grease: WWT plant effluent, acidified to pH 2	1 l	$\sim$2 l	Test range is 5 to 1000 mg/liter extractable material
Sample, Total Phosphorus: WWT plant effluent of known mg/liter P so dilutions can be assigned	50 ml	$\sim$1.5 l	Test range is 0.01 to 1.00 mg/liter P

4. Instructional Resources

a. Introduction

Most training institutions will make the fullest possible use of pre-existing instructional resources. The purpose of this section is to describe the nature, sources, and availability of instructional resources suggested for use with this course.

1) The Instructional Package Worksheets (IPWs) in Part II of this Manual and the student reference text refer to a number of different instructional resources. These include:

   a) The student reference text itself;

   b) Audiovisual training aids; and

   c) Supportive references

2) Instructional resources are discussed in this section from the viewpoint of the sources of the materials:

   a) Resources developed by United States Environmental Protection Agency (U.S. EPA)

   b) Resources developed by other sources; and,

   c) Resources already in possession of the training institution conducting this course.

b. Instructional Resources Developed by U.S. EPA

1) Student Reference Text and Staff Guide for the course "Effluent Monitoring Procedures Nutrients":

   a) While present supplies last, a sample copy (can be duplicated) is available on specific request to:

National Training and Operational Technology Center
ATTN: Training Information Clerk
U.S. Environmental Protection Agency
Cincinnati, Ohio 45268

   b) NTOTC has negatives of the Text and Guide which are available for temporary loan on request of a sponsoring Agency wishing to duplicate the materials.

c) NTIS can supply copies of the Text and Guide. A paper copy of the Text (PB-261-290/AS) costs $9.75. Contact NTIS for the identification number and cost of a paper copy of this Guide.

A microfiche copy of either the Text or Guide is also available from NTIS at $3.00 each.

U.S. Department of Commerce
National Technical Information Service
5285 Port Royal Road
Springfield, Virginia 22151

d) Your state agency may be able to arrange a direct supply of the texts and/or guides. Before ordering/reproducing materials, you might consult with your state agency about this possibility.

2) Audiovisual Training Aids Developed for the Course:

- a) What is available, according to topic:
  - (1) Use of a Spectrophotometer
    - (a) X-8, Slides, unassembled-16
    - (b) XT-51, Tape/Slide Unit, "Use of the Spectronic 20 Spectrophotometer", 10 minutes, 35 slides
    - (c) TC-57, Videocassette Tape Unit "Use of a Spectrophotometer", 21 minutes
  - (2) Preparation of Calibration Graphs
    - (a) X-9, Slides, unassembled-6
  - (3) Determination of Total Phosphorus (asP) or of Orthophosphate (asP), Single Reagent Method
    - (a) OT-7, Overhead Projecturals-7
    - (b) XT-44, Tape/Slide Unit, "Determination of Phosphorus", 15 minutes, 65 slides
  - (4) Determination of Chemical Oxygen Demand
    - (a) OT-8, Overhead Projecturals-4
    - (b) X-7, Slides, Assembled-34

(5) Determination of Total Kjeldahl Nitrogen

   (a) OT-9, Overhead Projecturals-9

   (b) Two Videocassette Tape Units are proposed for development by Charles County Community College

(6) Nitrogen, Ammonia Determination (wet)

   (a) XT-67, Tape/Slide Unit, "Determination of Ammonia Nitrogen", 8 minutes, 45 slides

(7) Determination of Nitrate-Nitrite Nitrogen and of Nitrate Nitrogen, Cadmium Reduction Method

   (a) OT-10, Overhead Projecturals-11

   (b) TC-58, Videocassette Tape Unit, "Preparation of the Cadmium Reduction Column", 21 minutes

(8) Determination of Oil and Grease

   (a) XT-56, Tape/Slide Unit, "Determination of Grease and Oil", 15 minutes, 54 slides

   (b) TC-55, Videocassette Tape Unit, Part I, "Basic Skills", 17 minutes and Part II, "Use of a Separatory Funnel", 13 minutes

   (c) TC-56, Videocassette Tape Unit, "Distillation Set Up", 15 minutes

(9) Determination of Ammonia by an Ammonia Selective Ion Electrode

   (a) X-10, Slides, unassembled-6

b) How to request loan of audiovisual training aids:

   (1) All items described in b.2)a) above are available on scheduled loan from NTOTC to institutions conducting this course. Requests should contain the information items on the "Request for Loan" form (next page). Send requests to NTOTC at the address given in b.1)a) above.

REQUEST FOR LOAN
AUDIOVISUAL INSTRUCTIONAL UNIT

Title and Catalog No. ______________________________

____

Intended Use: __________________________________

____

Preferred Date of Use: ____________________________

Alternate Date: _________________________________

BORROWER'S NAME ______________________________

Title ____

Organization ___________________________________

Address ______________________________________

____
(Zip)

Phone Number (include
Area Code): ________________________

There is no charge for use of the Audiovisual Instructional Units. However, the *BORROWER* assumes financial responsibility for the value of all loaned equipment and instructional materials.

Unless special arrangements are made with the loaning office, units should be returned within two weeks. Return the unit by *REGISTERED, CERTIFIED* or *INSURED MAIL IMMEDIATELY* after use.

EPA-171 (Cin)
(8-74)

(2) It is urged that materials desired from NTOTC for a specific course offering be requested in a single, consolidated communication. This will give greatest assurance of a well-coordinated response. Because these requests ordinarily will cover a number of different items, telephonic requests should not be made.

(3) Requests should be timely. To assure effective delivery in time for use in the course, requests should be received at NTOTC at least 45 days prior to the course date. The Center will, in turn, make every effort to assure that the requested materials are delivered to the requesting institution several days prior to the start of the course in which they are to be used. This will permit review and practice by the instructional staff for the most effective use of such resources.

(4) It is expected that all borrowed resources be returned to the Center within two weeks after completion of the course in which they are used.

(5) With returned borrowed training resources, it is requested that the user provide the Center with an evaluation of the training resource(s) used. In this manner the experience of users can be a factor in continuous improvements and responses to problems in using the resources. All reports on use of such resources should include the number of students with whom the material was used.

3) Supportive References:

a) Manual: EPA-EMSL, "Methods for Chemical Analysis of Water and Wastes". This is the reference source of all the methods presented in this course in the Effluent Monitoring Procedure (EMP) format. Address requests for a copy to:

U.S. EPA
Office of Technology Transfer
Industrial Environmental Research Laboratory
Cincinnati, Ohio 45268

b) AV Catalog: NTOTC, "Audiovisual Instructional Units". This is a catalog of slide-tape instructional units developed by the Center. Although not developed specifically for this course, several of the units are on course topics and might be useful supplementary material. Address requests for a copy to NTOTC at the address given in b. 1) a) above.

c) IRIS: A Water Quality Instructional Resources Information System has been developed through an EPA training grant. The "Master Reports" contain availability information and descriptions for 2300 entries of instructional and resource materials. These are printed matter, slides, films, slide-tape units and video tapes developed by varied sources for training personnel involved in all aspects of water quality assessment and control. Thus, IRIS serves as an information source of available, water quality training resources covering a wide range of subjects.

The user does not need data processing equipment to use the system. Four of the commonly used elements of IRIS (Users Manual, Tables, Master Report, Subject Index) are currently available.

(1) While present supplies last, a set is available to qualifying educational institutions and training agencies from NTOTC at the address given in b.1)a) above.

(2) Paper copies can be ordered as a set of four volumes from NTIS (PB-262-223/AS, Set, 1120 pp. "Water Quality Instructional Resources Information System, Volumes I through IV) for $31.00. The NTIS address is given in b.1)c) above. Microfiche copies cost $12.00 per set.

(3) Paper or microfiche copies of individual volumes can also be ordered from NTIS at the address given in b.1)c) above using this information:

(a) PB-262-224/AS, 99 pp., "Water Quality Instructional Resources Information System, Volume I-Users Manual" @ $5.00. (microfiche $3.00)

(b) PB-262-225/AS, 96pp., "Water Quality Instructional Resources Information System, Volume II-IRIS Tables" @ $5.00 (microfiche $3.00)

(c) PB-262-226/AS, 494 pp., "Water Quality Instructional Resource Information System, Volume III - Identification Number Master Report" @ $12.50 (microfiche $3.00)

(d) PB-262-227/AS 431 pp., "Water Quality Instructional Resources Information System, Volume IV - Subject Index" @ $11.75 (microfiche $3.00)

c. Instructional Resources Developed by Other Sources

1) Minimum technical references which should be in possession of the institution include:

a) "Standard Methods for the Examination of Water and Wastewater" (14th ed), APHA, AWWA, WPCF. Available from Publications Office, American Public Health Association, Inc., 1015 18th Street, NW, Washington, D. C., 20036.

b) "Annual Book of Standards" Part 31, "Water", 1975. Available from American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103.

2) Audiovisual and Other Training Aids

   a) A wide variety of training resources is listed in the EPA sponsored publication, "Water Quality Instructional Resources Information System" (IRIS), cited in b. 3) c) above.

   b) Information on sources of the listed items is provided.

   c) Training institutions having information about training resources applicable to this course, which are not currently listed in IRIS, are invited to relay this information to the Director, National Training and Operational Technology Center. Such resources, as applicable, will be made known to other organizations which could benefit from their use.

d. Instructional Resources Already in Possession of the Training Institution

1) Many training organizations prefer to develop their own texts and audiovisual training resources.

2) To the extent that these resources can be released for free reproduction and use by others, institutions are invited to make such resources available to other training organizations.

3) The National Training and Operational Technology Center is prepared to serve as a focal point for making information about such resources widely available, provided that copyright or other restrictions on reproduction do not limit availability of such materials.

   a) Before encouraging other training institutions to use such resources in relation to this Course, elements of EPA will:

      (1) Review the training resources to determine whether the instruction is consistent with existing laws, regulations, and Agency policy;

      (2) Review the resource for technical validity and educational quality.

   b) Materials found suitable by EPA would be recommended to other institutions known to be presenting this Course.

4) All training resources referred to NTOTC as available for use by others will be added to the overall inventory listing cited in IRIS, b. 3) c), above. It is hoped that a mutually supportive activity in this area will, in time, result in:

   a) making IRIS a diversified, total resource system for training materials which will be of the highest technical quality;
   b) offering training institutions a wide variety of types of training resources;
   c) reducing the amount of duplication of effort that so often results from lack of information on what is available, from what sources, and how obtained.

C. Secretarial Support

The key role of the office worker(s) designated "Course Secretary" cannot be overemphasized.

This function has many elements including being the right arm of the Course Coordinator, being Course Registrar, being Course Secretary and being a "Course Watchdog" to give the alarm when essential milestone stages of course planning and preparation are being overlooked.

In this section, these elements are considered under the headings: Course Records and Record-keeping, Suggested Student Registration Procedures and Printed and Reproduced Materials - Summary.

1. Course Records and Record-keeping

a. General Considerations

1) Complete, detailed, and accurate records should be established for each course presentation. Each course record will be a separate file. In addition to the individual course files, it may be necessary to establish a finder-system for locating the records of individual students.

2) Response to Inquiries about Former Students

a) Students enroll in this course in order to acquire necessary knowledge and skills to perform the self-monitoring procedures required for municipal effluents.

b) In many, if not all cases, satisfactory completion of this course will be a factor in the accreditation of individuals to perform the analyses and measurements required for compliance with NPDES Permits.

c) It is anticipated that numerous inquiries from former students and from regulatory agencies will be addressed to the training institution. Typical requests for information may include any or all of the following:

(1) Verification of attendance and satisfactory completion of training;

(2) Identification of the specific analyses and measurements covered in the course, as well as designation of the method which was taught;

(3) Quality of student performance in the course;

(4) Documentation of any specific analyses, tests, or measurements in which the student did not meet the required standard of performance, and the nature of such failure; and

(5) Documentation of any other events which made the student unusual. This could be a record of exceptionally high performance, or it could be a record of any specific difficulty which arose in connection with the student, within or outside the scheduled training activities.

3) Reports

Most training institutions require submission of periodic reports on progress and achievements. It is safe to predict that management or cognizant regulatory agencies from time to time will call for information not provided in routine reports. If such demands are to be met, complete course records will be the most reliable source for such information.

4) Retention of Records

The length of time course record files should be retained is uncertain, and must be determined by each institution.

Institutions having a system of archives for inactive files may find it convenient to retain course records in active office files for approximately two years, then retire them to archives storage. Institutions not having archives storage probably should retain the complete file on each course presentation for at least five years.

b. Contents of Course Files

1) In the planning and development stage, and until completion of each course presentation, course records are kept most effectively in two sections.

These are:

a) A file folder, kept in the filing cabinet or in the desk of the Course Secretary; and,

b) A student record notebook, usually a 3-ring binder, kept on the Course Secretary's desk, or in a convenient bookcase.

Both sections of the Course files should be maintained by the Course Secretary, and should be made available to other staff members under rigid controls providing for direct examination and immediate return. After completion of the course, the two sections can be combined in a single large file packet for future retention.

2) The file folder is best suited for such records as:

   a) Copies of all correspondence, memoranda, and records of telephonic conferences related to course planning and development;

   b) Copies of course schedules;

   c) Records of equipment and supply acquisition for the course, through purchase or through loan (with information and records on return to owner);

   d) Records of staff assignments, classroom and laboratory reservations;

   e) Copy of course announcement and/or description (See pages 12-7 and 8);

   f) Sample record copies of all routine informational material sent to students accepted for training; (See pages 12-15 through 18);

   g) Records of arrangements for travel of personnel and transportation of equipment and supplies, arrangements for field facilities, and other records pertaining to a field course; and

   h) Course evaluation commentaries by Course Coordinator and other staff members as appropriate.

3) The student record notebook is best suited as a vehicle for all records and copies of communications related to individual students. This may be organized effectively in a 3-ring notebook, containing separator sheets with alphabetical tabs. The personal records of each student will be retained under the alphabetical tab corresponding with his last name. In the student record notebook may be found:

   a) At the front (before the "A" of the series of tabbed dividers):

      (1) A registration summary sheet showing record of standard communications with each accepted student, fees paid, etc... (See page 12-19);

      (2) A waiting list summary sheet showing record of standard communications with each student placed on a waiting list prior to the course due to early maximum enrollment. (See page 12-20)

      (3) A non-attendance summary sheet showing record of students who applied for admission but could not be admitted for lack of qualification, or due to an already-filled class. This summary also is used to identify applicants who applied for admission, who were admitted, and who failed to appear without due explanation ("no shows"). (See page 12-21);

(4) A summary sheet recording student performance (acceptable or not acceptable) for each of the units of instruction in the course. (Such a summary worksheet has not been developed at the writing of this Guide).

b) In the alphabetical section of the student record notebook, each student's personal record will contain such items as:

(1) The application for admission to training (See pages 12-9 and 10);

(2) Copies of all correspondence with the student (See pages 12-11 through 14), except for the routine local information sheets (See pages 12-15 through 18), one set of which is kept in the loose file folder;

(3) Record copies of student quizzes, data sheets, and other individual records of class performance provided by Instructor. (See pages 12-25 through 39).

(4) Copy of the certificate awarded at end of course which is a record of the measurements completed by the student in a satisfactory manner. (See page 12-24);

(5) Documentation of any information about the student judged to be of possible future concern or inquiry. (An example of such a sheet had not been developed at the time of writing this Guide).

2. Suggested Student Registration Procedures

a. Introduction

1) Purpose

Formal registration and enrollment procedures are intended to assure that:

a) The class consists of students for whom the training is intended and designed;

b) The accepted students meet minimum knowledge and skills required for reasonable assurance of satisfactory completion of the course;

c) Accepted students are provided with adequate pre-training information so that they will make their personal arrangements and travel schedules to assure arrival at the appointed time and place, with full participation throughout the program of training;

d) The size of the class is in accordance with the course plan; and

e) Those not accepted for training are provided with suitable advice which can lead to future admission.

2) Alternative Approaches to Registration

Three different approaches to registration are considered here, though only the first is described in detail. Most details of the second and third identified alternatives can be inferred through study of the first alternative. The three approaches considered are:

a) Registration by priority of receipt of application;

b) Registration by comparative evaluation of all applicants; and

c) Registration for special course offering at request of another organization. Students are nominated by the requesting organization and are accepted without further evaluation of entry-level qualifications by the training institution.

b. Registration by Priority of Receipt of Application (Recommended)

1) Applicant

a) Receives course announcement;

b) Completes application and secures internal approvals as required in his own organization;

c) Mails application to registration office of the institution conducting the training.

2) Course Secretary

   a) Receives application;

   b) As received, checks status of registration for availability of class space, and makes appropriate notation on the application or on attached transmittal slip;

   c) Depending on b), records application either in the registration summary, or the waiting list summary, in front of student notebook;

   d) Delivers application to Course Coordinator.

3) Course Coordinator

   a) Evaluates the student application and the space availability situation;

   b) Directs the Course Secretary by notation on the application to:

      (1) Accept the applicant, or

      (2) Notify the applicant of closed class enrollment, or

      (3) Refer the applicant to the "Basic Laboratory Skills" course before entry into this course;

   c) Prepares a special letter for non-admissable applicants (or memorandum record of other form of communication with applicant) which sets forth the reasons why the applicant is being rejected. Because of the compulsory nature of the self-monitoring tests on municipal effluents for NPDES Permit compliance, rejection of a bona fide applicant may be a very serious matter, and should be handled with care.

4) The Course Secretary

   a) Prepares a standard letter or special letter as directed, and obtains signature of Course Coordinator:

   b) Places a file copy of the letter and the application in the appropriate place under the alphabetical tab section of the student record notebook;

   c) Mails the original letter to the applicant, and records the the date of mailing in the appropriate place in the summary records at the front of the student notebook.

   d) Approximately 30 days before the course, mails to each accepted applicant a standard communication, consisting of

      (1) A form letter of welcome to the course, including information on starting and closing dates and hours of the course, directions and how to proceed to the classroom area, and related information (See page 12-14); and

(2) Local information helpful to outside visitors, such as hotel/motel information, local transportation and schedules, a schematic map of the area, and related information. (See pages 12-15 through 18);

e) Records the mailing of the standard packet on the registration summary sheet.

f) When applications are still being accepted within 30 days before the start of the course, includes the general informational material with the letter of acceptance for admission.

g) On the first day of the course:

(1) Obtains a detailed registration card from each student. Some institutions may dispense with this record, though it can be of value in report preparation (See page 12-22);

(2) Prepares any registration tallies required by requesting organization(s) and/or administrative regulations. (See page 12-23);

(3) Prepares a class roster of those in attendance, distributes to class, staff, and keeps a permanent record copy in course files;

(4) Records any "no shows" (applicants accepted for training but who did not appear) on the student non-attendance summary record sheet. (See page 12-21)

c. Registration by Comparative Evaluation of all Applicants

1) Applicant

a) Receives course announcement;

b) Completes application and secures internal approvals as required in his own organization;

c) Mails application to registration office of the institution conducting the training.

2) The Course Secretary

a) Receives the applications;

b) Records receipt of application in a summary record in student record notebook;

c) Files application in student notebook;

d) Sends standardized letter acknowledging the application, and briefly explaining the registration procedure, with assurance that decision on admission will be announced not less than 30 days prior to start of the course;

e) Delivers all applications to the Course Coordinator , approximately 35 days before start of the course.

3) The Course Coordinator

a) Reviews and evaluates all applications;

b) Selects students to be admitted for training;

c) Directs Course Secretary to send appropriate standardized letters and information packets as described in b. above;

d) Special note should be taken of the particular attention which should be given to rejected applicants. See b., 3), c). above.

4) The Course Secretary

a) Sends communications;

b) Prepares records and student files as described in b.4) above.

d. Registration for Special Course Offering

Here a requesting organization has designated a student body which it wishes to have trained. It is the duty of the training institution to provide the requesting organization with admission standards for the course. It becomes the duty of the requesting organization to screen its candidates for conformance to these standards, and to provide the training institution with the names of the students to be trained. The requesting organization usually notifies the students.

1) For record purposes, it is best that students complete a course application form, though it will not be evaluated as in b. and c. above.

2) On receipt in the training institution, the Course Secretary makes the necessary entries showing record of receipt and class composition. Files are kept in the usual way. Approximately 30 days before the course, the individual standardized welcome and information packets are sent to students in the usual way. The training organization follows its usual practices in preparation of records, rosters, and any other data required for reports.

3. Printed and Reproduced Materials - Summary

a. General Information

In addition to the student reference text, standardized letters and administrative forms/materials, presentation of this course also requires calculation forms, laboratory data sheets, and graph forms which must be prepared in quantity by the Course Secretary.

1) In the following summarizing table, all of the standardized materials noted above are identified and supported with additional information on due date, the number to be prepared (for a class of 18 students), and the ultimate fate of the materials in permanent course records. Institutions offering this course may find it necessary to add to or to modify these standardized materials. It is suggested that plans to do so be noted on the summarizing table, with samples or examples provided on separate pages.

2) A sample or example of each item listed (except the student reference text) is shown following the summarizing table.

   a) Samples can be copied directly, if meeting requirements of the training institution.

   b) The examples are shown in recognition that a corresponding item probably will be needed by the training institution, but probably will have to be modified to fit the situation.

b. Responsibilities for Printed and Reproduced Material

1) Course Coordinator

   a) Reviews the administrative materials for conformance to the requirements of the regulatory authority;

   b) Makes modifications as necessary to the samples and examples provided in this Guide;

   c) Decides upon and designs any additional administrative documents or records needed; and

   d) Provides the Course Secretary with complete identification of material to be copied directly or to be modified, and also provides samples of any new material required for course administration.

2) Instructors

   a) Review all materials identified for the procedures for which they have instructional responsibility;

   b) Design new supportive instructional material as required; and

c) Provide the Course Secretary with complete information on material to be copied directly or to be modified, and also provide samples of any new material required for student instruction.

3) Course Secretary

a) Receives from Course Coordinator and Instructors identification of existing materials, samples of modified and new material;

b) Adds to the summarizing table, in the appropriate locations, the identifying information, together with the supporting information on due date, quantity, confidentiality, and ultimate fate of any new or revised material designed by Course Coordinator or by Instructors;

c) Reproduces, or arranges reproduction of, the needed course materials so that they will be available for use at the time and place required.

c. Special Warnings

1) All staff members should be particularly alert to adjustments in "Due Date" which must be made when the course is conducted in the field, if training equipment and supplies must be shipped to the course site.

2) Preparation of these training materials is a potential source for great difficulty in course development and presentation. Few activities in course planning and development require a greater amount of effective teamwork among all staff members. The greatest problem here is one of timing.

a) All staff members must provide necessary information and samples of new or modified materials with adequate lead time to meet "Due Dates." The amount of lead time is not specified here; this will vary from one institution to another.

b) The Course Secretary must be diligent in advising Course Coordinator and Instructors of impending logistic problems if delays occur in submission of materials, and must give prompt attention to printing or reproduction of needed materials when delivered by staff members.

c) The author of this guide ruefully confesses that the worst and most frequent breakdowns in this area usually are the result of belated delivery of needed material from Instructional Staff to the Course Secretary.

SUMMARY OF REQUIRED PRINTED/REPRODUCED MATERIAL

Description	Lesson	When Needed	Number to be Prepared	Confidential ?	Permanent Record?	Remarks
Administrative						
Course Announcement	-	6 months before course	Indeterminate	No	1 copy	Distribute to target group 6 months before course.
Course Description	-	6 months before course	Indeterminate	No	1 copy	Same as announcement. Can be used in conjunction with chronological course listings.
Application for Admission	-	6 months before course		No	No	Usually part of course announcement. May be separate sheet.
Prerequisite Verification	-	6 months before course	Indeterminate	No	1 copy	Attached to application form
Standard Letter: Acceptance	-	5 months before course	100	No	No	Copies will show up in student files.
Standard Letter: Standby: Full Class - Waiting List	-	5 months before course	100	No	No	Copies will show up in student files.
Standard Letter: Referral to Basic Lab Skills Course	-	5 months before course	100	No	No	Copies will show up in student files.
Standard Letter: Welcome and Local Information: Hotels/Motels, Transportation Schedule, Schematic Area Map Classroom Location	-	30 days before course	100	No	1 copy	In Course file folder.
Registration Summary Record	-	5 months before course	1	No	Yes	In Registrar's three-ring notebook

Description	Lesson	When Needed	Number to be Prepared	Confidential ?	Permanent Record?	Remarks
Administrative (Cont'd.)						
Waiting List Summary	-	90 days before course	1	No	Yes	In Registrar's three-ring notebook
Non-attendance Summary	-	First day of Course	1	No	Yes	In Registrar's three-ring notebook
Trainee Registration Card	-	First day of course	20	No	Yes	Institution's Records
Registration Tally and Class Roster	-	First day of course	As required	No	Yes	In course file folder
Course Certificates	-	Final day of course	18	No	Yes	In student file
Student Performance Summary Record	-	Final day of course	18	Yes	Yes	In Registrar's three-ring notebook
Classroom/Laboratory Use of a Spectrophotometer						
Example Data Sheet	1 of 1	Day 1	20	No	Yes	Duplicate completed, corrected sheet and keep in students' files.
Summary of Student Performance	-	Final day of course	18	No	Yes	Keep in students' files.
Preparation of Calibration Graphs						
Graph Paper	1 of 1	Day 1	20	No	Yes	Duplicate completed, corrected graph and keep in students' files.
Summary of Student Performance	-	Final day of course	18	No	Yes	Keep in students' files.

Description	Lesson	When Needed	Number to be Prepared	Confidential ?	Permanent Record?	Remarks
Total Phosphorus or Orthophosphate						
Example Data Sheet	2 of 5	Day 2	20	No	Yes	Duplicate completed, corrected sheet and keep in students' files.
EMP Calibration Graph	4 of 5	Day 2	20	No	Yes	Duplicate completed, corrected graph and keep in students' files.
Summary of Student Performance	-	Final day of course	18	No	Yes	Keep in students' files.
Chemical Oxygen Demand						
Example Data Sheet I	1 of 6	Day 2	20	No	Yes	Duplicate completed, corrected sheet and keep in students' files.
Standardization of FAS Calculation Sheet	2 of 6	Day 3	20	No	Yes	Duplicate completed, corrected sheet and keep in students' files.
Summary of Student Performance	-	Final day of course	18	No	Yes	Keep in students' files.
Total Kjeldahl Nitrogen						
Example Data Sheet	1 of 3	Day 3	20	No	Yes	Duplicate completed, corrected sheet and keep in students' files.
Absorbance Data Sheet	2 of 3	Day 3	20	No	Yes	Duplicate completed, corrected sheet and keep in students' files.
EMP Calibration Graph	2 of 3	Day 3	20	No	Yes	Duplicate completed, corrected sheet and keep in students' files.
Summary of Student Performance	-	Final day of course	18	No	Yes	Keep in students' files.

Description	Lesson	When Needed	Number to be Prepared	Confidential ?	Permanent Record?	Remarks
Nitrogen Ammonia						
Example Data Sheet (either for titration or Nesslerization)	1 of 1	Day 3	20	No	Yes	Duplicate completed, corrected sheet and keep in students' files.
Summary of Student Performance	-	Final day of course	18	No	Yes	Keep in students' files.
Nitrate + Nitrite Nitrogen						
Example Data Sheet	7 of 11	Day 4	20	No	Yes	Duplicate completed, corrected sheet and keep in students' files.
EMP Calibration Graph for NO_2 + NO_3-N	8 of 11	Day 4	20	No	Yes	Duplicate completed, corrected sheet and keep in students' files.
EMP Calibration Graph for NO_2-N	8 of 11	Day 4	20	No	Yes	Duplicate completed, corrected sheet and keep in students' files.
Summary of Student Performance	-	Final day of course	18	No	Yes	Keep in students' files.
Oil and Grease						
Example Data Sheet	1 of 1	Day 5	20	No	Yes	Duplicate completed, corrected sheet and keep in students' files.
Summary of Student Performance	-	Final day of course	18	No	Yes	Keep in students' files.
Ammonia Electrode						
Summary of Student Performance	-	Final day of course	18	No	Yes	Keep in students' files.

(EXAMPLE ANNOUNCEMENT)

ANNOUNCING
USEPA SPONSORED INSTRUCTOR TRAINING FOR PERMIT COMPLIANCE

The **National Training and Operational Technology Center** has developed a series of "packaged" courses for training municipal treatment plant personnel how to sample, measure, and analyze their wastewater discharges so as to comply with NPDES Permit requirements. Instructional materials for each course have been prepared in modular form, with each module containing detailed step-by-step procedures for the performance of a specific monitoring task. A description of each course so far developed appears below.

The **National Training and Operational Technology Center** provides tuition-free courses for instructors engaged in this type of training in Federal, State, and Local water pollution control programs, and in educational institutions. In these courses the student-instructors perform each self-monitoring task. In addition, they become thoroughly familiar with the organization and recommended use of the instructional materials; with training aids and other technical assistance available from USEPA for this type of training; with the planning and implementation of intensive training programs; with recommended instructional techniques; and with USEPA regulatory requirements for NPDES self-monitoring. These latter subjects are dealt with in depth only in course 164.1 which is an 8 day offering for student-instructors. It is recommended that interested instructors attend this course prior to any of the others.

164.1
Effluent Monitoring Procedures:
Basic Parameters for Municipal Wastewaters
5 days
(8 days for student-instructors)

This course is intended for municipal wastewater treatment plant technicians and others engaging in analysis of wastewater treatment plant effluents for compliance with requirements of discharge permits issued under the National Pollutant Discharge Elimination System. Upon completion of training, the student will be able to collect samples, perform the tests, and report results for BOD, fecal coliforms, pH, suspended solids, residual chlorine, and measure flow by weirs and parshall flumes. Laboratory test procedures conform to Agency-approved methods as promulgated in the Code of Federal Regulations.

164.2
Effluent Monitoring Procedures:
Metals Analysis
5 days

This course is designed for wastewater treatment plant technicians who will be responsible for performing selected metals analyses in treatment plant effluents. Participants will perform selected metals analyses including: boron, copper, iron, mercury, sodium, and zinc. Classroom instruction is limited to information about performing these analyses and reporting the results. Most of the time is given to laboratory experiences for the trainee who uses detailed, stepwise procedures to analyze typical samples. Procedures will be in conformance with Agency-approved methods as promulgated in the Code of Federal Regulations.

164.3
Effluent Monitoring Procedures:
Nutrients
5 days

This course is designed for municipal wastewater treatment plant technicians who are responsible for self-monitoring of nutrient concentrations in treated effluents to comply with requirements of discharge permits issued under the National Pollution Discharge Elimination System. Upon completion of the course, participants will be able to perform selected analysis for Chemical Oxygen Demand; the Nitrogen Series (Total, Ammonia, Nitrate, Nitrite); Total Phosphorus. The test procedures conform to Agency-approved methods as promulgated in the Code of Federal Regulations.

164.5
Effluent Monitoring Procedures:
Flow Measurement & Sampling Techniques
5 days

This course is designed for the treatment plant operator or technician who is required to monitor effluent discharges under a National Pollutant Discharge Elimination System (NPDES) Permit, and who has had little or no previous experience in collection of wastewater samples, or in measuring wastewater flows. Following classroom discussions and demonstrations, participants measure flows and collect samples at a wastewater treatment plant using both manual methods and automatic devices; and also measure effluent parameters on-site; using portable field instrumentation.

☆ U.S. GOVERNMENT PRINTING OFFICE [illegible] 757-005/0005

APPLICATION FOR INSTRUCTOR TRAINING

I am interested in attending the following training course or courses in Cincinnati, Ohio:

1. Effluent Monitoring Procedures: Basic Parameters for Municipal Wastewaters (164.1) July 11-20, 1977 ☐
2. Effluent Monitoring Procedures: Metals Analyses (164.2) June 13-17, 1977 ☐
3. Effluent Monitoring Procedures: Nutrients (164.3) August 1-5, 1977 ☐
4. Effluent Monitoring Procedures: Flow Measurement and Sampling Techniques (164.5) June 27 - July 1, 1977 ☐

(For additional offerings of these courses, call the NTOTC Registrar – 513/684-7501)

Organization in which you will be providing instruction: ____________________

Your Name ______________________________ Phone ____________

Business Address ______________________________

(SAMPLE COURSE DESCRIPTION)

EFFLUENT MONITORING PROCEDURES: NUTRIENTS (Course 164.3)

5 Days

November 3-7, 1975 June 28-July 2, 1976

This course is designed for the treatment plant operator or technician who is required to monitor effluent discharges under a National Pollutant Discharge Elimination System (NPDES) Permit, and who has had little or no previous experience in wastewater analysis.

The course includes procedures for measuring Total Phosphorus (as P), Chemical Oxygen Demand, Kjeldahl (Total) Nitrogen, Ammonia (as N), Organic Nitrogen by difference of Kjeldahl N and Ammonia N, Nitrate-Nitrite (as N), Nitrite (as N), Nitrate (as N) by difference of Nitrate-Nitrite N and Nitrite N, and Oil and Grease. The course also includes procedures for related skills--using a spectrophotometer and preparing a calibration graph.

During the course, the student will perform an approved analytical procedure for each of the measurements. At the conclusion, he will be given a certificate verifying which measurements he performed in a satisfactory manner.

PRE-REQUISITES

Self-Monitoring Procedures: Course I - Basic Laboratory Skills or equivalent experience is pre-requisite for the course. A "Student Skills Checklist," signed by the applicant's employer, must be submitted before an application for the course can be processed.

June, 1976 TUITION: $175.00

SAMPLE *SAMPLE*

COURSE APPLICATION FORM

1. **Name of Applicant:** Mr. / Miss / Mrs. ______________________________
(last) (first) (middle initial)

2. **Course desired:**
Course Title ____________________ Course No. __________
Place where given ____________________ Dates __________

3. **Previous Courses Attended:**
Course Title ____________________ Dates __________
Course Title ____________________ Dates __________
Course Title ____________________ Dates __________

4. **Sponsor or Employer:** ______________________________
(name of organization or firm)
____
(street address)
____
(city) (state) (zip code) (telephone)

5. **Mailing address of applicant:**
(if different from above) ______________________________
(street address)
____
(city) (state) (zip code) (telephone)

6. **Job Duties:** (Briefly describe your present position)
____
____
____
____
____

7. **Education:** Last year of school completed ____________________

8. **Experience:** Total years in wastewater treatment plant work ____________________

9. **Student Skills Checklist:** This checklist must be submitted before Application can be processed.

____
signature of supervisor (where applicable) title

____
signature of applicant date

(SAMPLE STUDENT SKILLS CHECKLIST)

Name ______________________________

Employer ___________________________

To assist us in processing applications, please check YES or NO for each of the following items:

	YES	NO
I have operated a laboratory gas burner	___	___
I have operated a laboratory hotplate/stirrer	___	___
I have operated an autoclave	___	___
I have operated a laboratory drying oven	___	___
I have used a vacuum source to filter liquids	___	___
I have used a desiccator	___	___
I have weighed items on an analytical balance	___	___
I have weighed items on a single pan balance	___	___
I have used a graduate to measure liquids	___	___
I have used a volumetric pipet to measure liquids	___	___
I have used a graduated (Mohr) pipet to measure liquids	___	___
I have used a pipet bulb to fill a pipet	___	___
I have used a volumetric flask to prepare solutions	___	___
I have used chromic acid to clean glassware	___	___
I have operated a laboratory safety shower	___	___
I have operated a laboratory eye washer	___	___
I have operated a fume hood	___	___
I have prepared 0.0375N potassium biiodate solution	___	___
I have made out labels for bottles of reagents	___	___
I have recorded a reading at a meniscus	___	___
I have titrated one solution against another to a color change end point	___	___
I have calculated the normality (N) of a solution	___	___
I have recorded laboratory data in a laboratory notebook	___	___
I have entered laboratory data on a pre-printed form	___	___
I have recorded information about samples on record sheets	___	___
I have located required purchase information in a catalog of laboratory equipment	___	___
I have written a purchase order for chemicals	___	___
Volume means space occupied by a solid, liquid, or gas	___	___
mg/l means milligrams per liter	___	___
1 kilogram equals 0.001 gram	___	___
1 inch equals 2.54 cm	___	___
1000 ml equals 1 liter	___	___
85 times 4.1 equals 42.5	___	___
7 minus 2 divided by 0.02 equals 250	___	___
3.26 rounded to the nearest tenth is 32.6	___	___
84.55147 rounded to the nearest thousandth is 84.551	___	___

(SAMPLE LETTER OF ACCEPTANCE FOR TRAINING)

(Institutional Letterhead)

(Date)

To

Dear (Name):

A reservation has been confirmed for your participation in the course "Effluent Monitoring Procedures: Nutrients" to be conducted at (address, including building and room identification if pertinent).

Formal class activities will begin promptly at 8:30 AM on Monday, (date) and the course will be completed by 12:30 PM on Friday, (date). Please arrange your travel schedule so that you will be in the classroom at the start of course activities on Monday and that you will not have to hurry your departure on Friday.

Information about local travel, transportation, and local hotels is enclosed for your assistance. We believe that you will wish to make your own hotel or motel reservations.

We look forward to seeing you at the course, and we will do everything in our power to make this course a pleasant and rewarding experience for you.

Sincerely yours,

(signature)
Course Coordinator

Note: If something develops which makes it impossible for you to attend the course, please telephone (number) or write this office immediately, in order that another applicant may be admitted to the course in your place. Please do not arrange for a substitute without first getting the approval of this office.

(SAMPLE STANDBY LETTER)

(Institutional Letterhead)

(Date)

To

Dear (Name):

We have received your application for admission to the course "Effluent Monitoring Procedures: Nutrients" to be conducted at (name of institution) during the period (date to date).

We would be most pleased to enroll you in this course, but by the time we received your application, all available positions in the class had been reserved. As you may know, we limit the class size to a fixed number in order to provide for the greatest possible amount of personal instruction during the course, and to provide each participant with the greatest possible opportunity for actual practice in the laboratory.

We have made a tentative reservation for you in the next offering of the course, which is scheduled to be given (dates). If this will be satisfactory to you, please write or call us within (number) days, so that we can confirm your reservation.

In the meantime, we have placed your name on the waiting list for the course dates which you requested. If a vacancy does become available, we will let you know immediately.

Sincerely yours,

(signature)
Course Coordinator

(SAMPLE LETTER OF REFERRAL)

(Institutional Letterhead)

(Date)

To

Dear (Name):

We have received your application for admission to the course "Effluent Monitoring Procedures: Nutrients," to be conducted at (name of institution) during the period (date to date).

Based on our review of your previous laboratory experience, we consider that it would be very doubtful whether this course would provide you with all the knowledge and skills you will require to perform the self-monitoring tests and measurements on your municipal wastewater effluents.

Accordingly, we are confirming your reservation in this course, subject to your first satisfactorily completing the course (title of "Basic Skills" course). This course will be conducted at (location) (dates). We have made a tentative reservation for you to attend this course. Please let us know if you can attend this offering of the course.

If you must delay taking the course (name of "Basic Skills" course) at this time, then it will be necessary to delay your acceptance in the course which you have requested.

In the course (name of "Basic Skills" course) you will learn many things not covered in the later course, including use of the analytical balance, preparation and standardization of laboratory reagents, care and maintenance of laboratory supplies and equipment, and related tasks.

We are most anxious to help you learn to perform all the tasks required for self-monitoring of your municipal wastewater effluents. Please let us know if you can come to both courses.

Sincerely yours,

(signature)
Course Coordinator

(SAMPLE LETTER OF WELCOME)

(Institutional Letterhead)

(Date)

TO: COURSE PARTICIPANTS --

We are looking forward to your participation in the course "Effluent Monitoring Procedures: Nutrients", scheduled for presentation at this Center during the period (date to date). If you find you cannot attend the course, please call us (telephone number).

To assist your planning and preparation for this course, the following items are enclosed:

1. List of hotels and motels
2. Information on local bus transportation and city map, (NOTE: If bus service is used to the Center, you must have exact fare of (amount) on on boarding bus).

On your arrival in the classroom you will be provided a course manual and related materials. Production schedules make it impossible to mail manuals to you in advance of course date.

The course will start at 8:30 AM on Monday, (date) in (room identification) and will close no later than 12:30 PM, Friday, (date). At the conclusion of the course, a certificate will be awarded verifying which analytical measurements you have performed in a satisfactory manner. Please arrange your travel schedule for after closing exercises. (Approximately (number) hours should be allowed for travel from (name of institution) to the airport.)

This course involves a considerable amount of work in the laboratory, using concentrated acids and bases. You will want to bring appropriate clothing.

(Name), of our staff, is serving as Course coordinator and will be available to assist you in solving any special problems you encounter while attending the course.

Should you have questions or desire assistance in any way, please do not hesitate to contact us.

Sincerely yours,

(signature)
Director
Institution Name

(EXAMPLE INFORMATION SHEET)

DOWNTOWN HOTELS

CINCINNATIAN HOTEL
6th & Vine Sts.
Cincinnati, Ohio 45202

Phone: 513/241-0180

Single	$6.45
Double	8.60
Twin	10.75

225 Rooms
Meeting Room 1 - Capacity 25
TV, restaurant adjoining

NETHERLAND HILTON HOTEL
35 W. 5th Street
Cincinnati, Ohio 45202

Phone: 513/621-3800

Single	$22.00
Double	30.00
Twin	34.00

Family plan, 800 rooms, TV, meeting rooms 15 - Cap. 70-1500, special Gov't. rates - $16.00 single, $23.00 double, $24.00 - twin.

TERRACE HILTON HOTEL
15 W. 6th Street
Cincinnati, Ohio 45202

Phone: 513/381-4000

Single	$23.00
Double	31.00
Twin	35.00

Family plan, 350 rooms, meeting rooms 4 - cap. 75-400, color TV, special Gov't. rates - $18.00 single, $25.00 double, $26.00 - twin.

STOUFFER's CINCINNATI INN
150 W. 5th Street
Cincinnati, Ohio 45202

Phone: 513/721-8600

Single	$25.00 - 28.00
Double	31.00 - 34.00
Twin	31.00

462 rooms, meeting rooms 11, cap. 50-550, swimming pool, cocktail lounge, sauna bath, color TV, Gov't. rates - $20.00 single, $26.00 double.

HOLIDAY INN*
8th & Linn Sts.
Cincinnati, Ohio 45203

Phone: 513/241-8660

Single	$22.00
Double	28.00

245 rooms, meeting rooms 4 - cap. 25-135 swimming pool, TV, 2 dining rooms, and bars, night club "Top of the Inn" (entertainment nightly).

NOTE: We recommend you checking the rate at the time you make your reservation in the event there has been a price increase.

These hotels and motels are listed for your information to assist you in planning for your accommodations during your stay in Cincinnati while attending our training course, and does not imply endorsement by the Office of Water Program Operations, U.S. Environmental Protection Agency.

* REQUIRES TRANSFER TO SECOND BUS.

BUS SCHEDULE (EXAMPLE INFORMATION SHEET)

BUS NO. 53 - AUBURN/CLIFTON

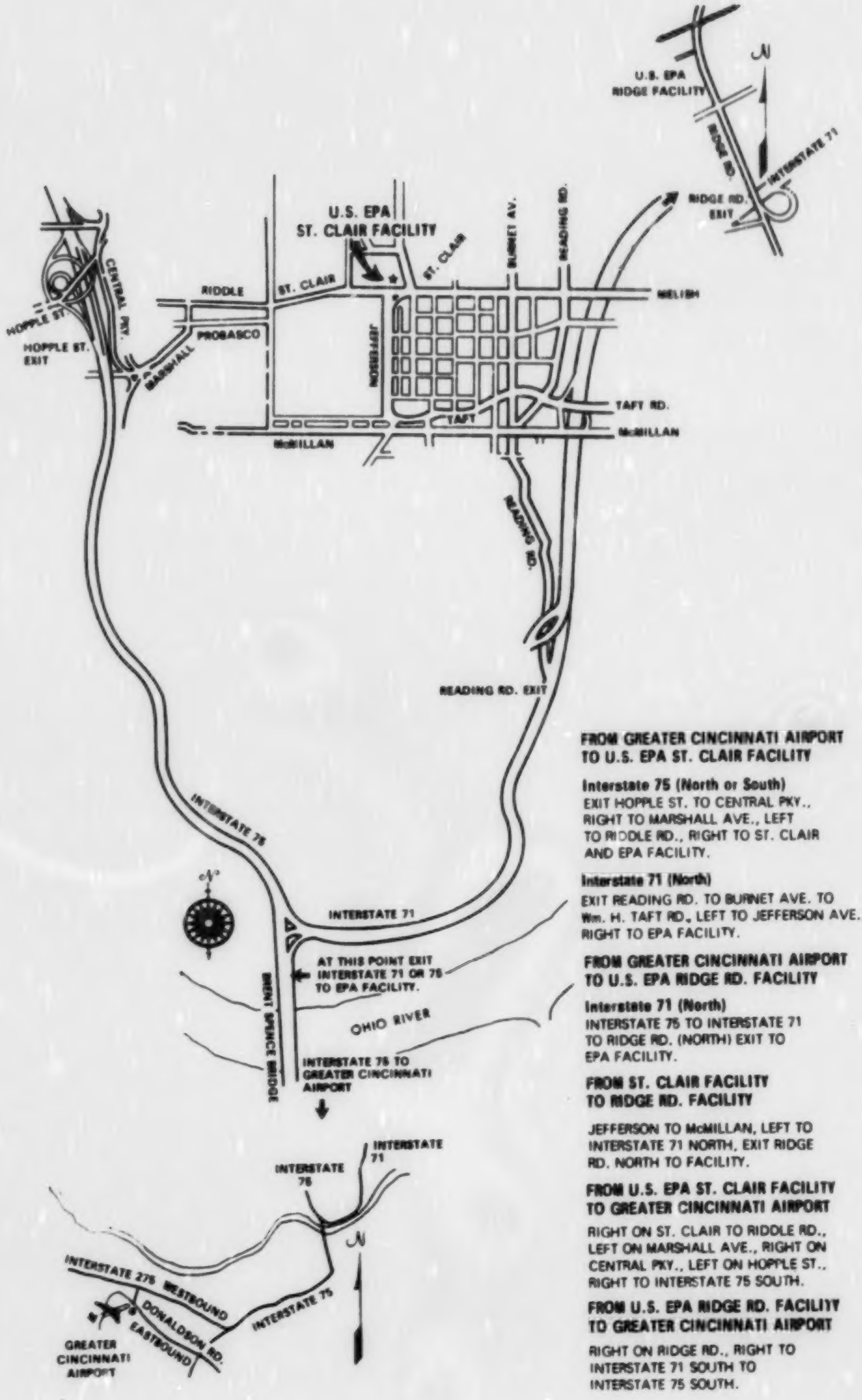
BUS NO. 78 - LOCKLAND/READING

BUS. NO. 53		BUS NO. 78	
Leave Sixth & Vine	Arrive St. Clair & Vine	Leave Sixth & Vine	Arrive St. Clair & Vine
AM	AM	AM	AM
7:18	7:36	7:09	7:22
7:41	7:59	7:27	7:40
8:03	8:21	7:44	7:57
8:25	8:43	8:04	8:17
8:46	9:04	8:40	8:53

BUS NO. 53		BUS NO. 78	
Leave St. Clair & Vine	Arrive Sixth & Vine	Leave St. Clair & Vine	Arrive Sixth & Vine
PM	PM	PM	PM
12:30	12:48	12:08	12:24
1:02	1:20	12:29	12:45
1:33	1:51	12:50	1:06
2:05	2:23	2:54	3:12
2:36	2:54	3:11	3:29
2:58	3:16	3:28	3:46
3:21	3:39	3:45	4:03
3:41	4:02	4:01	4:19
4:04	4:25	4:17	4:35
4:28	4:49	4:27	4:51
4:52	5:13	4:49	5:07

NOTE: The above two routes are the most direct routes to the Environmental Research Laboratory, 26 W. St. Clair Street, Cincinnati, Ohio. Information has been provided by the Queen City Metro. Times selected are those most apt to be used by students attending NTC Training Courses. Arrival times are approximate, and will vary because of road and traffic conditions.

(EXAMPLE INFORMATION SHEET)


U.S. EPA
RIDGE FACILITY
RIDGE RD.
INTERSTATE 71
RIDGE RD.
EXIT
U.S. EPA
ST. CLAIR FACILITY
ST. CLAIR
BURNET AV.
READING RD.
MELISH
RIDDLE
ST. CLAIR
HOPPLE ST.
HOPPLE ST.
EXIT
CENTRAL PKY.
MARSHALL
PROBASCO
JEFFERSON
TAFT RD.
TAFT
McMILLAN
McMILLAN
READING RD.
READING RD. EXIT
INTERSTATE 75
INTERSTATE 71
AT THIS POINT EXIT
INTERSTATE 71 OR 75
TO EPA FACILITY.
BRENT SPENCE BRIDGE
OHIO RIVER
INTERSTATE 75 TO
GREATER CINCINNATI
AIRPORT
INTERSTATE
75
INTERSTATE
71
INTERSTATE 275 WESTBOUND
EASTBOUND
DONALDSON RD.
INTERSTATE 75
GREATER
CINCINNATI
AIRPORT
N

FROM GREATER CINCINNATI AIRPORT TO U.S. EPA ST. CLAIR FACILITY

Interstate 75 (North or South)
EXIT HOPPLE ST. TO CENTRAL PKY., RIGHT TO MARSHALL AVE., LEFT TO RIDDLE RD., RIGHT TO ST. CLAIR AND EPA FACILITY.

Interstate 71 (North)
EXIT READING RD. TO BURNET AVE. TO Wm. H. TAFT RD., LEFT TO JEFFERSON AVE., RIGHT TO EPA FACILITY.

FROM GREATER CINCINNATI AIRPORT TO U.S. EPA RIDGE RD. FACILITY

Interstate 71 (North)
INTERSTATE 75 TO INTERSTATE 71 TO RIDGE RD. (NORTH) EXIT TO EPA FACILITY.

FROM ST. CLAIR FACILITY TO RIDGE RD. FACILITY

JEFFERSON TO McMILLAN, LEFT TO INTERSTATE 71 NORTH, EXIT RIDGE RD. NORTH TO FACILITY.

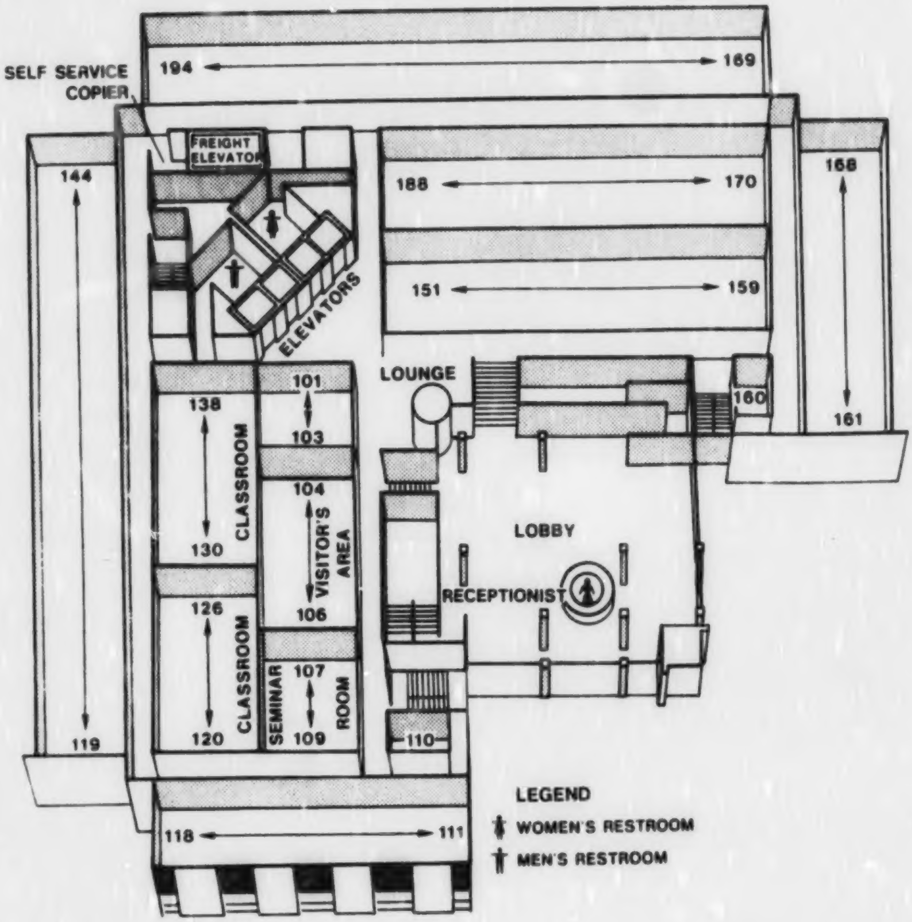
FROM U.S. EPA ST. CLAIR FACILITY TO GREATER CINCINNATI AIRPORT

RIGHT ON ST. CLAIR TO RIDDLE RD., LEFT ON MARSHALL AVE., RIGHT ON CENTRAL PKY., LEFT ON HOPPLE ST., RIGHT TO INTERSTATE 75 SOUTH.

FROM U.S. EPA RIDGE RD. FACILITY TO GREATER CINCINNATI AIRPORT

RIGHT ON RIDGE RD., RIGHT TO INTERSTATE 71 SOUTH TO INTERSTATE 75 SOUTH.

(EXAMPLE INFORMATION SHEET)


194
169
SELF SERVICE COPIER
FREIGHT ELEVATOR
144
119
188
170
151
159
168
161
ELEVATORS
LOUNGE
160
101
103
138
130
CLASSROOM
104
106
VISITOR'S AREA
126
120
CLASSROOM
107
109
SEMINAR ROOM
110
LOBBY
RECEPTIONIST
118
111
LEGEND
WOMEN'S RESTROOM
MEN'S RESTROOM

First Floor

(SAMPLE REGISTRATION SUMMARY)

(Course Title)

REGISTRATION

INDIVIDUAL	WORD REC'D.	APPL. REC'D.	ACCEPT. SENT	COURSE INFO. SENT

(SAMPLE WAITING LIST SUMMARY)

WAITING LIST

INDIVIDUAL	WORD REC'D	APPL. REC'D	ACTION TAKEN	

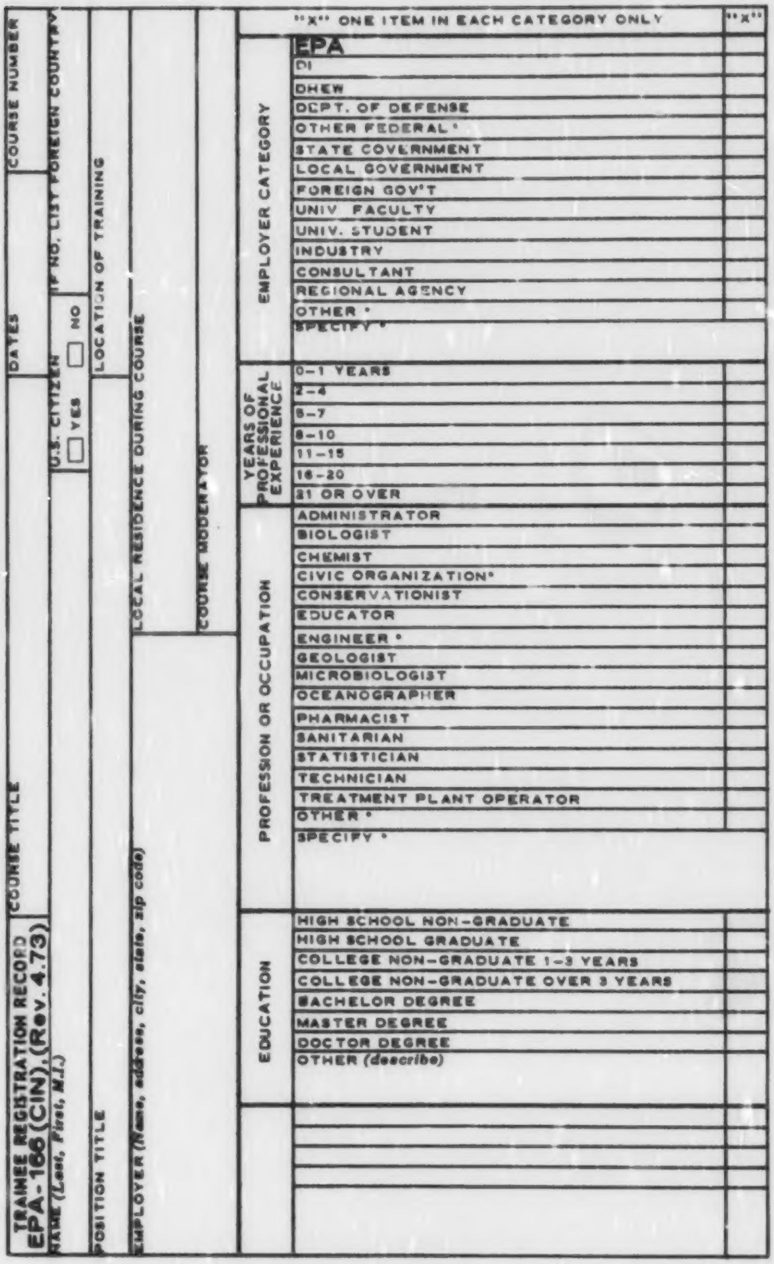
(SAMPLE NON-ATTENDANCE SUMMARY)

Course Title

Dates

Not Admitted Lack of Space	No Shows

(EXAMPLE REGISTRATION CARD)



TRAINEE REGISTRATION RECORD
EPA-166 (CIN), (Rev. 4.73)

COURSE TITLE

DATES

COURSE NUMBER

NAME (Last, First, M.I.)

U.S. CITIZEN ☐ YES ☐ NO

IF NO, LIST FOREIGN COUNTRY

POSITION TITLE

LOCATION OF TRAINING

EMPLOYER (Name, address, city, state, zip code)

LOCAL RESIDENCE DURING COURSE

COURSE MODERATOR

	"X" ONE ITEM IN EACH CATEGORY ONLY	"X"
EMPLOYER CATEGORY	EPA	
	DI	
	DHEW	
	DEPT. OF DEFENSE	
	OTHER FEDERAL *	
	STATE GOVERNMENT	
	LOCAL GOVERNMENT	
	FOREIGN GOV'T	
	UNIV. FACULTY	
	UNIV. STUDENT	
	INDUSTRY	
	CONSULTANT	
	REGIONAL AGENCY	
	OTHER *	
	SPECIFY *	
YEARS OF PROFESSIONAL EXPERIENCE	0-1 YEARS	
	2-4	
	5-7	
	8-10	
	11-15	
	16-20	
	21 OR OVER	
PROFESSION OR OCCUPATION	ADMINISTRATOR	
	BIOLOGIST	
	CHEMIST	
	CIVIC ORGANIZATION*	
	CONSERVATIONIST	
	EDUCATOR	
	ENGINEER *	
	GEOLOGIST	
	MICROBIOLOGIST	
	OCEANOGRAPHER	
	PHARMACIST	
	SANITARIAN	
	STATISTICIAN	
	TECHNICIAN	
	TREATMENT PLANT OPERATOR	
	OTHER *	
	SPECIFY *	
EDUCATION	HIGH SCHOOL NON-GRADUATE	
	HIGH SCHOOL GRADUATE	
	COLLEGE NON-GRADUATE 1-3 YEARS	
	COLLEGE NON-GRADUATE OVER 3 YEARS	
	BACHELOR DEGREE	
	MASTER DEGREE	
	DOCTOR DEGREE	
	OTHER (describe)	

(EXAMPLE REGISTRATION TALLY)

COURSE SUMMARY RECORD
EPA 165 (CIN). (Rev. 7.73)

LOCATION OF TRAINING	
COURSE DIRECTOR	
COURSE MODERATOR	
NO. OF TRAINEES	
COURSE TITLE	
DATES	
COURSE NUMBER	

ITEM	TALLY	TOTAL	
EPA			EMPLOYER CATEGORY
DI.			
DHEW			
DEPT. OF DEFENSE			
OTHER FEDERAL			
STATE			
LOCAL			
FOREIGN GOV'T			
UNIV. FACULTY			
UNIV. STUDENT			
INDUSTRY			
CONSULTANT			
REGIONAL AGENCY			
OTHER			
0-1 YEARS			YEARS OF PROFESSIONAL EXPERIENCE
2-4			
5-7			
8-10			
11-15			
16-20			
21 OR OVER			
ADMINISTRATOR			PROFESSION OR OCCUPATION
BIOLOGIST			
CHEMIST			
CIVIC ORG.			
CONSERVATIONIST			
EDUCATOR			
ENGINEER			
GEOLOGIST			
MICROBIOLOGIST			
OCEANOGRAPHER			
PHARMACIST			
SANITARIAN			
STATISTICIAN			
TECHNICIAN			
TREAT PLANT OPER			
HS NON-GRAD			EDUCATION
HS GRAD			
COL 1-3 YEARS			
COL OVER 3 YEARS			
BACHELOR DEGREE			
MASTER DEGREE			
DOCTOR DEGREE			
EPA			
PAID			

BY STATE OR COUNTRY OF EMPLOYMENT		
AREA	TALLY	TOTAL
ALABAMA		
ALASKA		
ARIZONA		
ARKANSAS		
CALIF.		
COLO.		
CONN.		
DELAWARE		
D. C.		
FLORIDA		
GEORGIA		
HAWAII		
IDAHO		
ILLINOIS		
INDIANA		
IOWA		
KANSAS		
KY.		
LA.		
MAINE		
MD.		
MASS.		
MICHIGAN		
MINN.		
MISS.		
MISSOURI		
MONTANA		
NEBRASKA		
NEVADA		
N. H.		
N. J.		
N. MEXICO		
NEW YORK		
N. C.		
N. D.		
OHIO		
OKLA.		
OREGON		
PENN.		
R. I.		
S. C.		
S. D.		
TENN.		
TEXAS		
UTAH		
VERMONT		
VIRGINIA		
WASH.		
W. VA.		
WISC.		
WYOMING		

(EXAMPLE CERTIFICATE)

(INSTITUTIONAL IDENTIFICATION)

APPROPRIATE

LOGO

This certifies that

THOMAS JONES

has completed the course

Effluent Monitoring Procedures: Nutrients (164.3)

and has performed the following measurements in a satisfactory manner:

MEASUREMENT OF TOTAL PHOSPHORUS ________________ INSTRUCTOR	MEASUREMENT OF AMMONIA NITROGEN (MACRO METHOD) ________________ INSTRUCTOR
MEASUREMENT OF CHEMICAL OXYGEN DEMAND ________________ INSTRUCTOR	MEASUREMENT OF NITRATE & NITRITE NITROGEN ________________ INSTRUCTOR
MEASUREMENT OF TOTAL KJELDAHL NITROGEN ________________ INSTRUCTOR	MEASUREMENT OF OIL & GREASE ________________ INSTRUCTOR

________________, 19__ to ________________, 19 __

(SIGNATURE)
Director, (Institution Name)

(SIGNATURE)
Course Coordinator

EFFLUENT MONITORING PROCEDURE: Use of a Spectrophotometer

EXAMPLE DATA SHEET

SPEC 20

A	C (mg/l)
0. _____	_____
0. _____	_____
0. _____	_____
0. _____	_____
0. _____	_____
0. _____	_____
0. _____	_____
0. _____	_____

A of sample = 0. _____

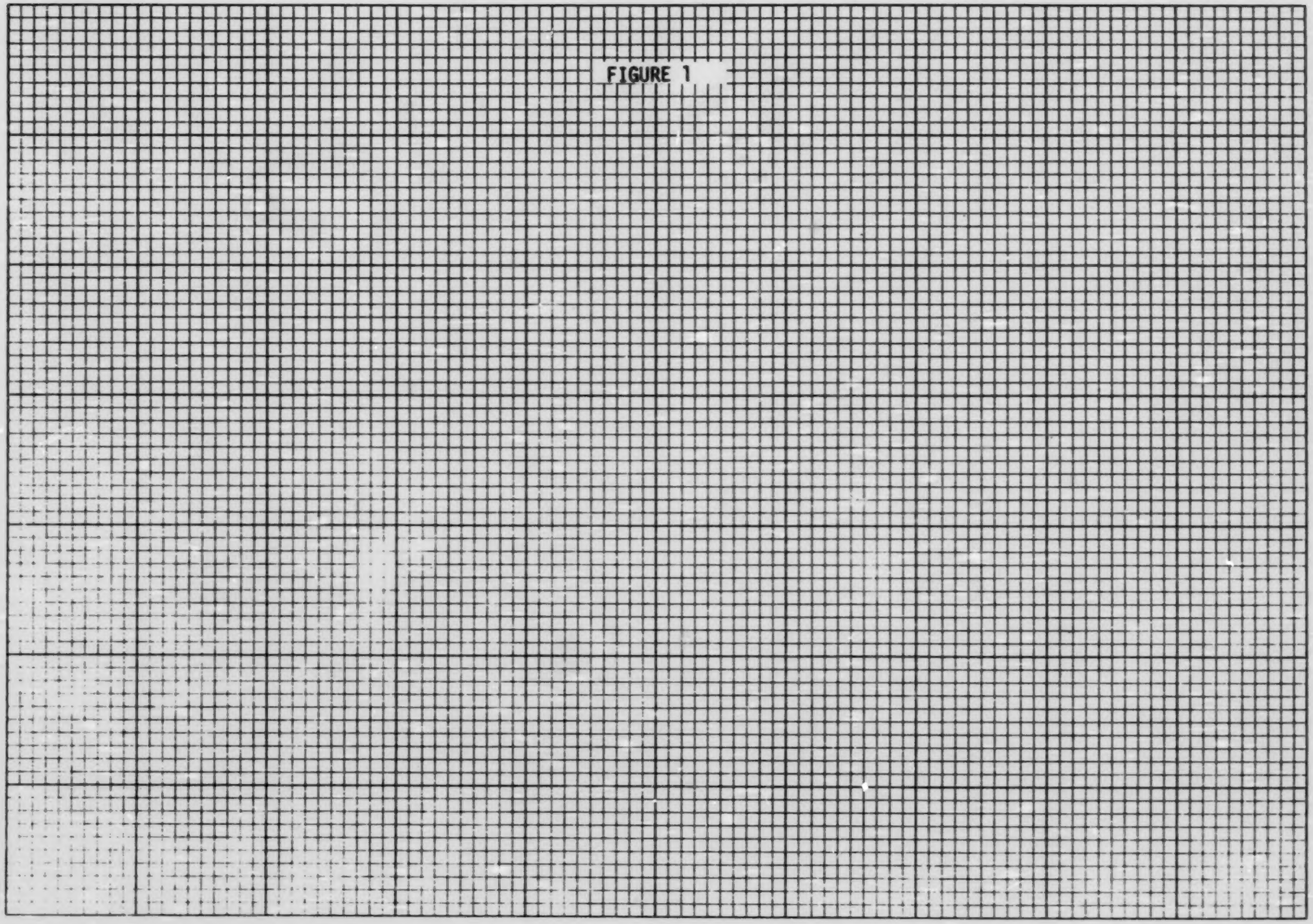


FIGURE 1

RECORDS AND REPORTS Section IX

EXAMPLE DATA SHEET FOR TOTAL PHOSPHORUS OR FOR ORTHOPHOSPHATE, mg/liter P

E.1c	Sampling Location	Final Effluent	
E.1c	Sample Identification	E.S. 1s.	
E.1c	Type of Sample	Grab or Composite	
E.1c	Date and Time Collected	1/17/75 9:00 a.m.	
E.1c	Sample Collector	Tom Sampler	
E.1c	Date and Time Analysis Began	1/17/75 9:30 a.m.	
N.5a	Analyst	Dick Analyst	

	Calibration Standards mg/liter P	Absorbance	Check Standards mg/liter P	Absorbance	Curve mg/liter P
J.2.1d L.3a L.8a	0.02				
	0.06				
	0.10		0.10		
	0.20				
	0.40				
	0.60				
	0.80		0.80		
	1.00				

	Amount of Sample Dilution	Absorbance	Curve mg/liter P	Dilution Factor	Final mg/liter P
E.4b J.2.2a M.2a N.2a N.4a					

EXAMPLE CALCULATIONS

	Amount of Sample Dilution	Absorbance	Curve mg/liter P	Dilution Factor	Final mg/liter P
N.1c	Straight Sample	off scale	-	1	-
	$\frac{1}{2}$ (25 ml sample / 50 ml total)	off scale	-	2	-
	$\frac{1}{5}$ (10 ml sample / 50 ml total)	0.3525	0.520	5	2.60
	$\frac{1}{10}$ (5 ml sample / 50 ml total)	0.1775	0.260	10	2.60
	$\frac{1}{20}$ (2.5 ml sample / 50 ml total)	0.0875	0.130	20	2.60

EFFLUENT MONITORING PROCEDURE: Determination of Total Phosphorus (as P) or of Orthophosphate (as P),
Single Reagent Method

RECORDS AND REPORTS

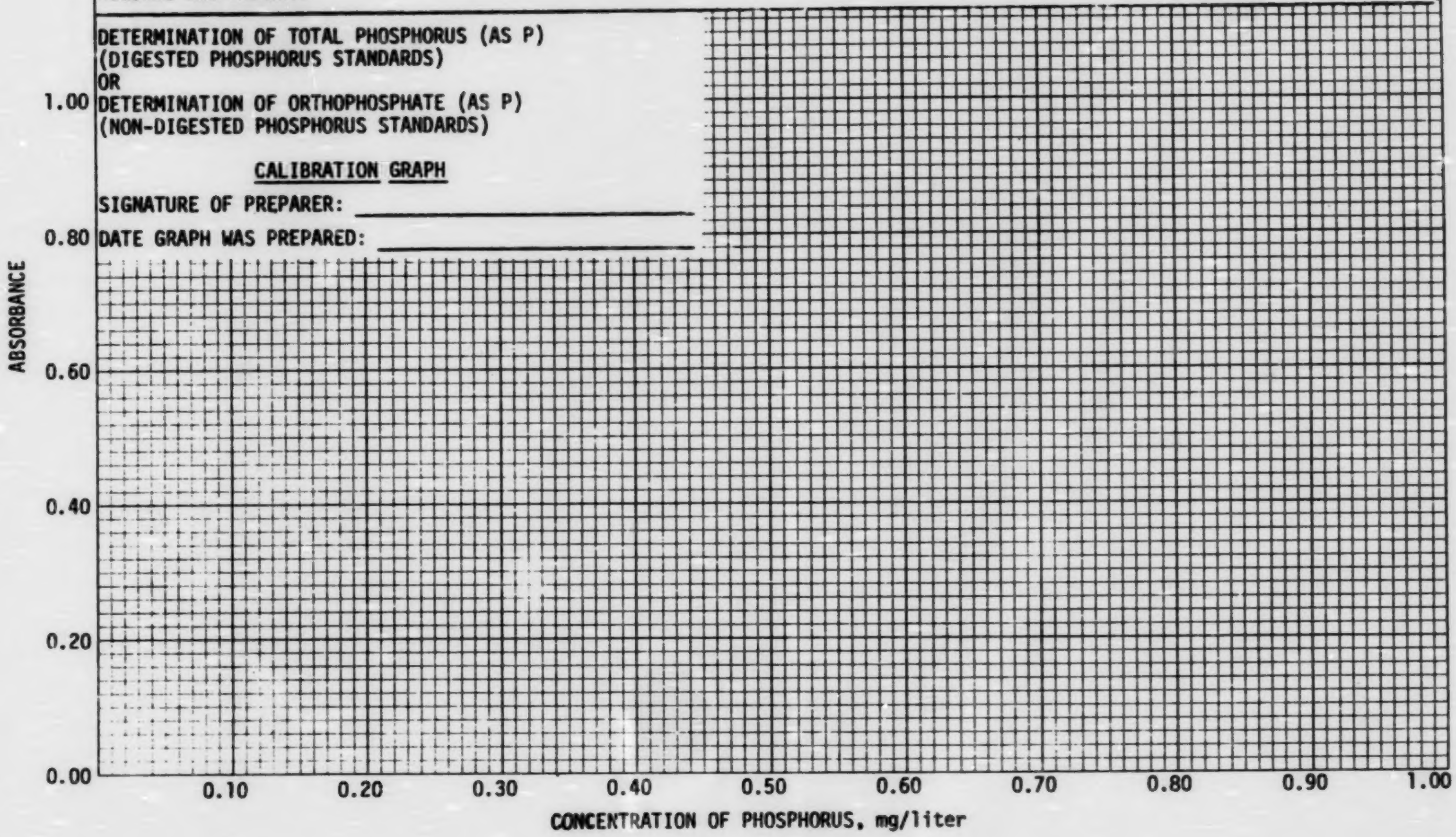
SECTION IX

DETERMINATION OF TOTAL PHOSPHORUS (AS P)
(DIGESTED PHOSPHORUS STANDARDS)
OR
DETERMINATION OF ORTHOPHOSPHATE (AS P)
(NON-DIGESTED PHOSPHORUS STANDARDS)

CALIBRATION GRAPH

SIGNATURE OF PREPARER: ______________________

DATE GRAPH WAS PREPARED: ______________________



Typical Laboratory Data Sheet for Chemical Oxygen Demand, mg/liter IX.SHEET I

Name of Plant____________________________________

A.5	Identification				Blank	EFF #1	1
A.5	Type (grab, composite)					Composite	2
A.5	Date and Time Collected					3/17/75 0600-1200	3
A.5	Sample Collector					Tom Sampler	4
F.35	Date and Time Boiling Began					3/17/75 1300	5
F.9	RECORD: S, ml Sample Used					50.0	6
H.15 H.22	ml FAS * at END of titration				38.55	20.00	7
H.7 H.18	ml FAS * at START of titration				15.00	5.00	8
H.23	A, ml FAS* used to titrate the Blank				23.55	23.55	9
H.16	B, ml FAS * used to titrate the Sample					15.00	10
J.3	SUBTRACT B (line 10) from A (line (9)					8.55	11
J.4	RECORD: N, normality of FAS * (Calculated on Standardization Sheet,C.29)					0.024	12
J.6	MULTIPLY ml Difference of FAS * (line 11) by Normality of FAS (line 12)					0.2052	13
J.8	DIVIDE 8000 by S, ml Sample Used (See line 6)					160	14
J.10	MULTIPLY line 13 by line 14					32.8320	15
J.12	ROUND OFF line 15 to the nearest whole number of mg/liter					33	16
J.13	Signature					Jim Analyst	17

* FAS means Ferrous Ammonium Sulfate Solution

CALCULATION FORMULA: COD, mg/liter = $\frac{(A-B)N \times 8000}{S}$

STANDARDIZATION OF FERROUS AMMONIUM SULFATE (FAS) SOLUTION IX SHEET II

				Flask 1	Duplicate	
C.31	ml FAS at END of titration			24.60	35.15	1
C.23	ml FAS at START of titration			14.00	24.60	2
C.33	ml FAS used for Standardization (SUBTRACT ml FAS at START on line 2 from ml FAS at END on line 1)			10.60	10.55	3
C.36	DIVIDE 0.250* by the ml difference on line 3 to a 4 decimal place answer.				0.0236	4
C.38	Normality of the FAS solution (ROUND OFF line 4 to 3 decimal places)				0.024	5
C.39	Date				3/17/75	6
C.40	Signature				Jim Analyst	7

* From the formula:

$$\text{Normality FAS} = \frac{(\text{10.0 ml potassium dichromate})\ (\text{0.025 N potassium dichromate})}{\text{ml FAS}}$$

	TRAINING GUIDE NOTE	REFERENCES/RESOURCES
	Typical Laboratory Data Sheet for Total Kjeldahl Nitrogen, mg/l Name of Plant____________________	
D.1.1c. E.1.1c.	Sampling Location____________________ Type of Sample____________________ Date and Time Collected____________________ Sample Collector____________________ Date and Time Analysis Began____________________ Analyst____________________ Method Used (Macro or Micro)____________________ ml. sample used....................................	____________________
D.4.12a E.4.12a	B. ml total distillate including boric acid (H_3BO_3) and dilution water................................	____________________
F.1.5c.	C. ml distillate taken for Nesslerization............	____________________
F.3.5a.	A. mg NH_3-N/50.0 ml, from curve.....................	____________________

Use this formula in calculating the results for the colorimetric method:

$$\text{TKN mg/l} = \frac{A \times 1000}{\text{ml sample}} \times \frac{B}{C}$$

(See pp. 5-31 and 5-32)

If Organic Nitrogen (mg/l) is needed and a separate ammonia analysis has been performed, use the following equation to determine this.

Since: TKN = Organic/N + Ammonia/N,

Then: Organic/N = TKN - Ammonia/N

Final Results

TKN mg/l____________________

NH_3-N, mg/l____________________

Org-N, mg/l____________________

	TRAINING GUIDE NOTE	REFERENCES/RESOURCES
F.2.6a	Values from Nesslerization Procedure	

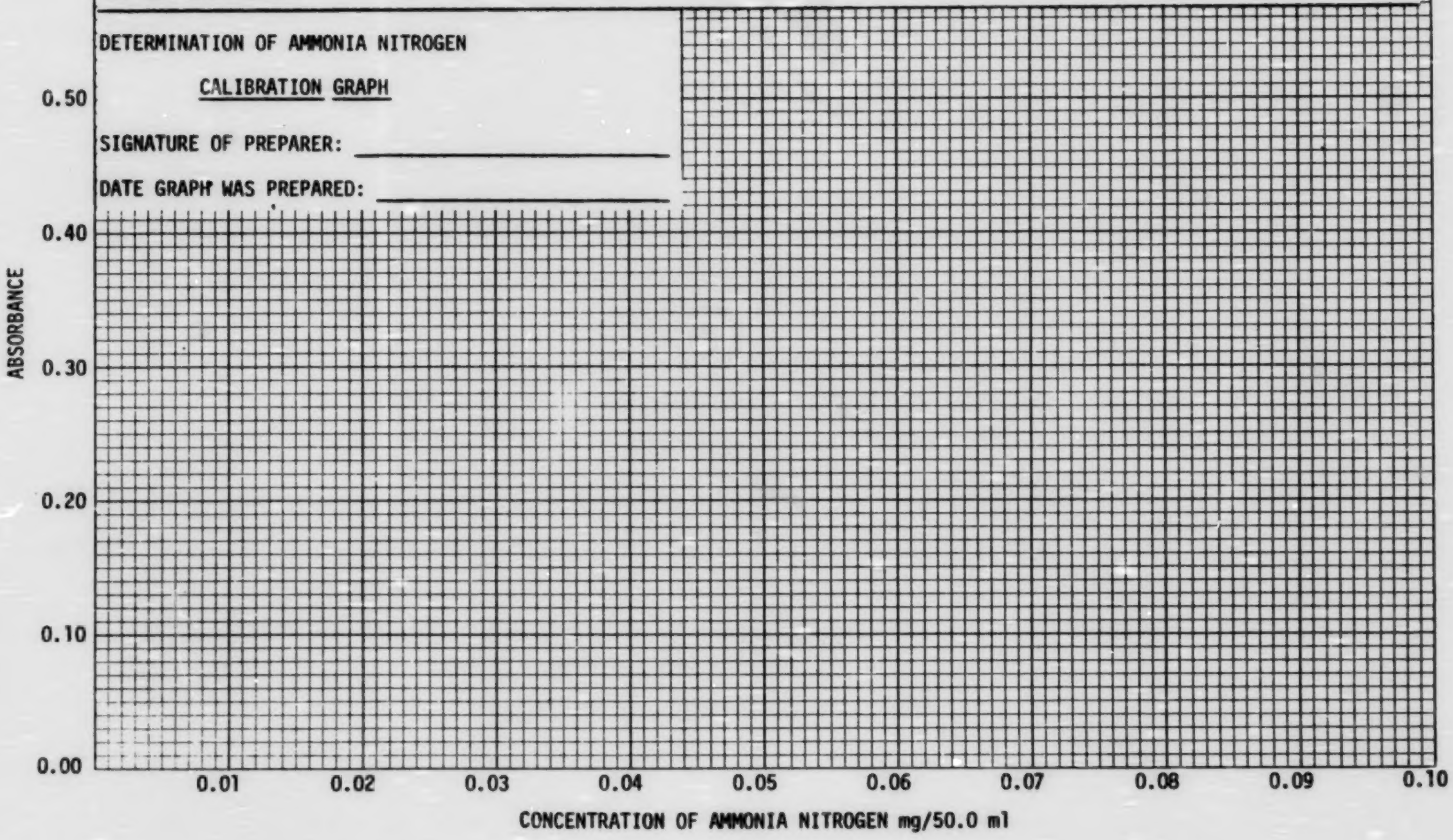
Tube #	Concentration mg hH_3-N/50.0 ml	Absorbance	Absorbance	Absorbance
1	0.0			
2	0.005			
3	0.010			
4	0.020			
5	0.040			
6	0.050			
7	0.080			
8	0.10			
9	Sample			
10	Sample			
11	Sample			

DETERMINATION OF AMMONIA NITROGEN

CALIBRATION GRAPH

SIGNATURE OF PREPARER: ______________________

DATE GRAPH WAS PREPARED: ______________________



LABORATORY DATA SHEET

Nitrogen, Ammonia Determination
(Sample contains 1.0 to 25.0 mg/l NH_3-N)

Sample No. ________ Date/Time Sampled ____________________ Sample Point ________

$$\frac{(\text{Sulfuric acid } 0.02N \text{ ml } \_\_\_\_\_\_)\ (0.28)(1000)}{\text{Sample ml } \_\_\_\_\_} = \_\_\_\text{mg/liter } NH_3\text{-}N$$

________________________ Date ____________

Analyst

LABORATORY DATA SHEET

Nitrogen, Ammonia Determination
(Sample Contains 0.05 to 1.0 mg/liter NH_3-N)

Sample No. ________ Date/Time Sampled ____________________ Sample Point ________

$$\frac{(\text{mg of } NH_3\text{-N}\_\_\_\_)(1000)}{(\text{Sample ml}\_\_\_)} \times \frac{(\text{Total Distillate* Collected ml}\_\_\_)}{(\text{Distillate Taken for Nesslerization ml}\_\_\_)} = \_\_\_\text{mg/l } NH_3\text{-N}$$

______________________________ Date __________

Analyst

*Include boric acid plus dilution water

EXAMPLE DATA SHEET

See Key on Page No. 7-47

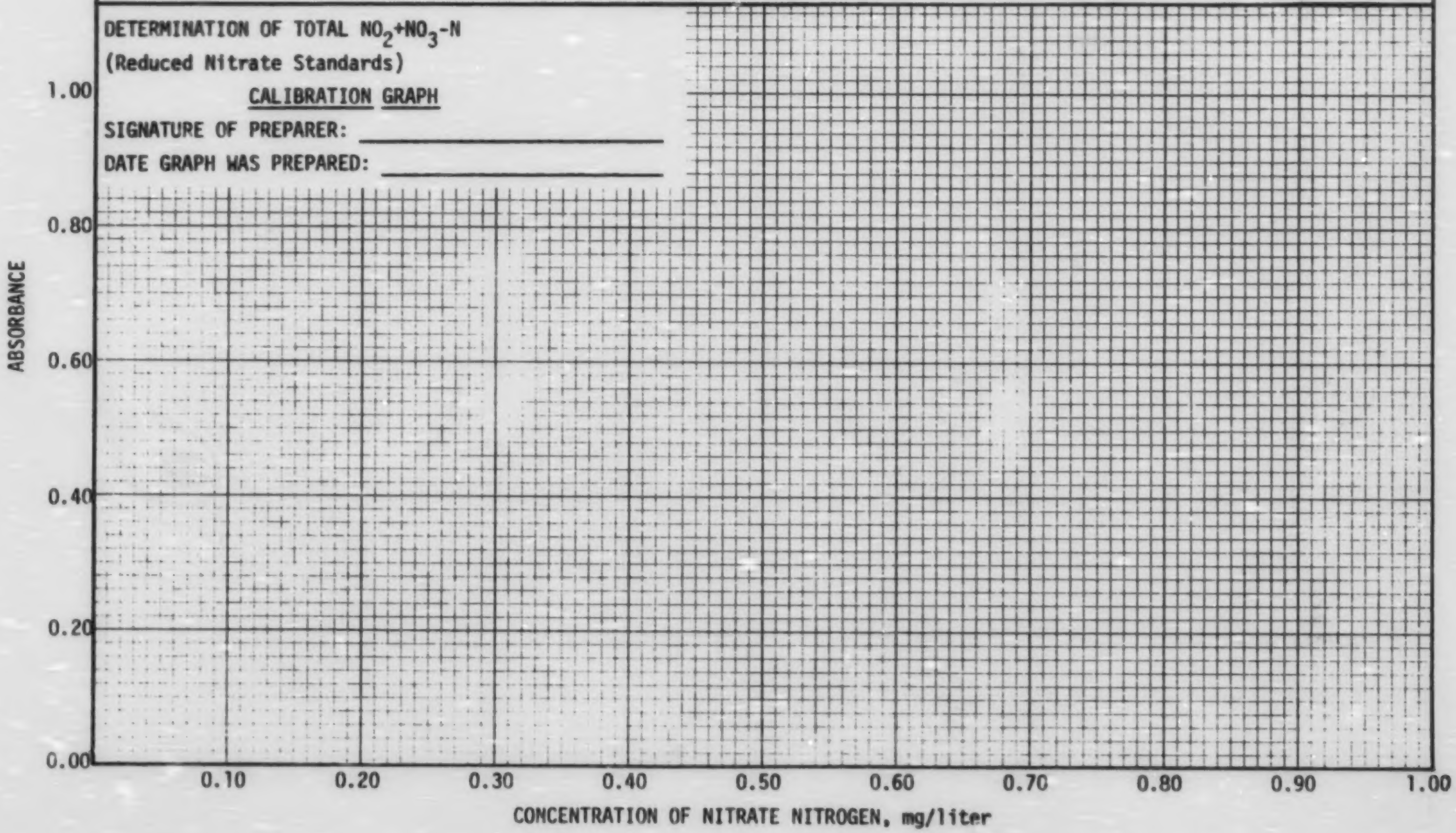
SAMPLE NUMBER	mg/liter TOTAL NO_2+NO_3-N (A)	ABSORBANCE OF TOTAL NO_2+NO_3-N (B)	mg/liter NO_2-N (C)	ABSORBANCE NO_2-N (D)	mg/liter NO_3-N (E)	mg/liter NO_3 (F)	mg/liter NO_2 (G)
Reduced Nitrate Working Standards							
2	0.05				0.05	0.22	
3	0.10				0.10	0.44	
4	0.20				0.20	0.89	
5	0.50				0.50	2.22	
6	1.00				1.00	4.43	
Reduced Sample(s)							
Non-reduced Nitrite Working Standards							
2			0.05				0.16
3			0.10				0.33
4			0.20				0.66
5			0.50				1.65
6			1.00				3.29
Non-reduced Sample(s)							

DETERMINATION OF TOTAL NO_2+NO_3-N
(Reduced Nitrate Standards)

CALIBRATION GRAPH

SIGNATURE OF PREPARER: ____________

DATE GRAPH WAS PREPARED: ____________



EFFLUENT MONITORING PROCEDURE: Determination of Nitrate-Nitrite Nitrogen and of Nitrate Nitrogen, Cadmium Reduction Method

RECORDS AND REPORTS

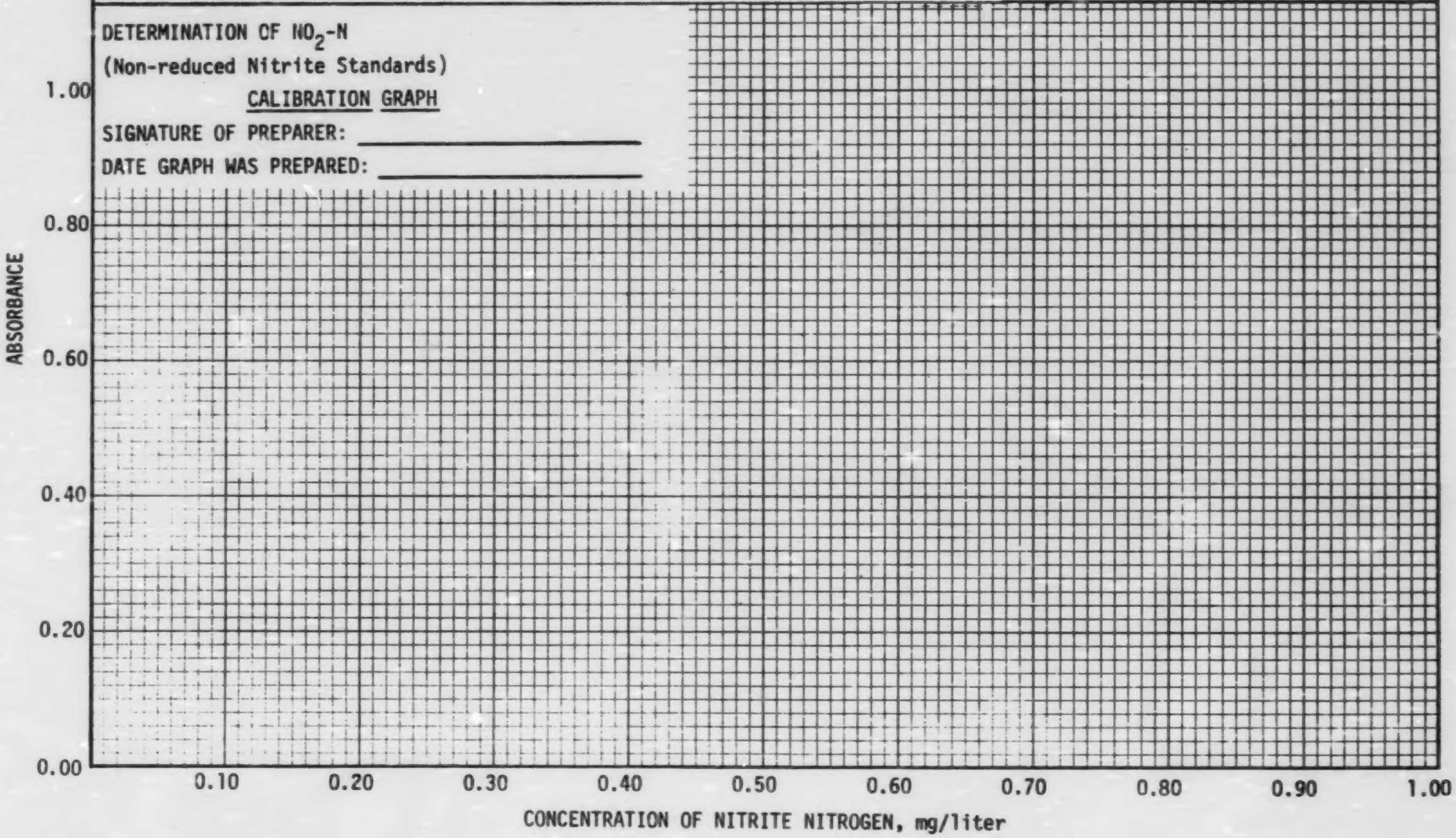
SECTION IX

DETERMINATION OF NO_2-N
(Non-reduced Nitrite Standards)

CALIBRATION GRAPH

SIGNATURE OF PREPARER: ____________________

DATE GRAPH WAS PREPARED: ____________________



EFFLUENT MONITORING PROCEDURE: Determination of Oil and Grease

Blank Determination

Weight of distilling flask after evaporation of the 100 ml of TF/D = E = ____________ grams

Weight of the empty distilling flask used to determine the blank = F = ____________ grams

Value of blank, D = E-F

Sample Determination

Weight of distilling flask + the oil/grease residue = A = ____________ grams

Weight of empty distilling flask used for the sample = B = ____________ grams

Volume of sample = C = ____________ milliliters

Milligrams of oil/grease residue per liter sample = $\frac{[(A-B)-D] \times 1000 \times 1000}{C}$

PART II - INSTRUCTIONAL PACKAGE WORKSHEETS

For each Effluent Monitoring Procedure (commonly termed "EMP" by Instructors) in the Student Reference Manual, there is an Instructional Package Worksheet (IPW) in this Guide. The Worksheet is for guidance to the Instructor for development of the subject matter covered in the course.

These Worksheets are not scripts. The Instructor will need to make extensive and detailed preparation in order to perform the assigned tasks effectively and efficiently. The Instructional Packages do provide a perspective on the background of each analytical procedure, lesson-by-lesson learning achievement levels the students should attain, an indication of available audiovisual and other instructional resources, and a recommended course of action in pre-course preparation and classroom/laboratory instruction.

Application of these Instructional Packages will help the Instructor to reduce the time required for planning and organizing a strategy of preparation and instruction. But time and effort are required for physical preparations for classroom and laboratory instruction; time and effort are required for rehearsals of Instructor performance in classroom and laboratory. These requirements never can be met by such a Course Guide as this; ultimately the Instructor is the key person in assuring that the student acquires the needed knowledge and skills.

A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES

for the

USE OF A SPECTROPHOTOMETER

as applied in

WASTEWATER TREATMENT FACILITIES
and in the
MONITORING OF EFFLUENT WASTEWATERS

INSTRUCTIONAL PACKAGE WORKSHEET

National Training and Operational Technology Center
Municipal Operations and Training Division
Office of Water Program Operations
U.S. Environmental Protection Agency

CH.IN.sp.ipw.2d.7.77

PART II INSTRUCTIONAL PACKAGE WORKSHEETS

A. Use of a Spectrophotometer

1. This skill is required in order to determine Phosphorus, Total Kjeldahl Nitrogen and Nitrate-Nitrite Nitrogen using the Effluent Monitoring Procedures (EMPs) for this course.

2. It was most practical to write the EMP for one instrument, the Bausch and Lomb* Spectronic 20-Spectrophotometer, and to use the manufacturer's instructions as the source of the procedure.

3. The steps in the EMP can be readily adapted for application to other instruments.

4. In the following Instructional Package Worksheet (IPW) for the EMP, the lesson plan is for processing a provided set of synthetic standards in a spectrophotometer, then a provided synthetic sample.

5. Each student is to process one set of standards and a sample made according to the directions in the IPW. The IPW equipment and reagent requirements are based on this assignment. If you choose a different assignment, adjust materials and quantities accordingly.

ERRATA: Please add this note in the EMP, Operating Procedure C at Step 7 in the third column:

7a. Always use a soft tissue to handle phototubes or to wipe off fingerprints.

---

* Mention of a specific brand name does not constitute endorsement by the Office of Water Program Operations, U.S. Environmental Protection Agency.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Use of a Spectrophotometer

UNIT OF INSTRUCTION:

LESSON NUMBER: 1 of 1

ESTIMATED TIME: 75 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: Several determinations required under the permit system involve the measurement of color intensity. The Bausch and Lomb Spectronic 20 spectrophotometer is one instrument which may be used to make the color intensity measurements.

ENTRY LEVEL BEHAVIOR: The learner must be able to:

1. Perform basic mathematical computations (addition, subtraction, multiplication and division).
2. Handle solutions of acids safely.
3. Clean laboratory glassware.

A. INSTRUCTIONAL OBJECTIVE

1. Terminal Behavior - The learner will exhibit proper technique while using the Bausch and Lomb Spectronic 20 spectrophotometer.

2. Conditions - The learner will have the use of the attached EMP, and all materials on the equipment list.

3. Accepted Performance - The use of proper technique will be judged by the instructor.

B. INSTRUCTIONAL RESOURCES

1. Available Media
    a. XT-51, Use of the Spectronic 20 Spectrophotometer, 10 minutes, tape and 35 slides.
    b. TC-57, Videocassette Tape Unit, "Use of a Spectrophotometer", 21 minutes, showing the steps listed in EMP Procedure B and a discussion of EMP Procedure C.
    c. NOTE: Either the Tape/Slide Unit or the Videocassette Tape Unit may be used.
    d. X-8, sixteen unassembled slides.

2. Suggested Media - None

C. INSTRUCTIONAL APPROACH (Sequencing)

1. Classroom (Using 16 unassembled slides)
    a. Discuss the concept of light being absorbed by a chemical solution.
    b. Define wavelength.
    c. Define nanometer.
    d. Define the Lambert-Beer law and the four terms in it. Absorbance and concentration values are needed to prepare a calibration graph. The calibration graph is the basis for quantitation in colorimetric measurements.
    e. Discuss how a series of colorimetric standards (e.g., as for the EPA phosphorus determination) would be prepared. Concentrations of the standards are known.

f. Mention that a Bausch & Lomb Spectronic 20 Spectrophotometer is one type of instrument which could be used to obtain absorbance values.
g. Show XT-51, Use of the Spectronic 20 Spectrophotometer, or the TV tape.
h. Discuss that it is now known how to obtain concentration and absorbance values.
i. Discuss the concept of a dilution factor and how it is used.
j. Have the learners read the Brief Description of Analysis.

2. Laboratory
   a. Have the learners begin the EMP at section A.3 by removing the "wrong" visible phototube from the Spectronic 20 (which you had previously inserted) following the EMP procedure. Have them next insert the "correct" infrared phototube and filter, and proceed with section B. The terms "wrong" and "correct" are used in reference to the methylene blue synthetic color standard. A break may be given during the warm-up period.
   b. Have the learners obtain absorbance values at 660 nm for a series of synthetic color standards prepared using methylene blue. (Before class, check that 660 is the proper wavelength for each Spectronic 20; i.e., the value may be a few nm more or less than 660.). In order to prepare the stock methylene blue solution, add a few drops of the indicator to about 300 ml of distilled water. The absorbance of the solution should be about 0.6. If it is not, add more distilled water or indicator as needed. Arbitrarily, mark this solution as having a concentration of 8.0 mg/l. Successive 50% dilutions will then give concentrations of 4.0, 2.0, 1.0, and 0.5 mg/l. Supply a set of methylene blue standards (including plain distilled water for 0.0 mg/l) with each Spectronic 20. Prepare a sample from the methylene blue such that the learners will have to dilute it 10 ml to 100 ml to obtain an "on-scale" absorbance reading; this will introduce the idea of using a dilution factor when the calibration graph is prepared. If desired, actual color standards could also be prepared using the phosphorus effluent monitoring procedure. An example data sheet for the methylene blue color standards is attached. Copies should be prepared and distributed to the learners. Note that the actual "concentration" of the diluted methylene blue sample will be determined after completion of the effluent monitoring procedure on the preparation of calibration graphs.

D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS

1. For each student:

   Laboratory apron

   Safety glasses

   One pen or pencil

   Notebook or data sheet

2. Shared:

   One Bausch and Lomb Spectronic 20 Spectrophotometer per three students

   One manufacturer's manual for each spectrophotometer

   Still, or other source of distilled water

One visible phototube (Bausch & Lomb catalog number 33-29-71) for each Spec 20
One infrared phototube (Bausch & Lomb catalog number 33-29-72) for each Spec 20
One infrared filter (Bausch & Lomb catalog number 33-29-18) for each Spec 20
Ten soft tissues (for wiping the cells)
One plastic squeeze distilled water bottle
Sink or 1 liter container for rinsing solutions
One 1 cm cell for each Spectronic 20
One 100 ml volumetric flask
One 10 ml volumetric pipet
One pipet bulb

E. IPW REAGENT REQUIREMENTS (Minimum amounts per group sharing a Spectronic 20)

One set of six methylene blue color standards. See C.2.b. above.
One bottle of methylene blue "sample" (to be diluted 10 to 100)

EXAMPLE
DATA SHEET
for
Methylene Blue Color Standards

Concentration in mg/l	Absorbance
0.0	0.____
0.5	0.____
1.0	0.____
2.0	0.____
4.0	0.____
8.0	0.____

Absorbance of diluted sample 0.____

Dilution factor = 10

A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES

for the

PREPARATION OF CALIBRATION GRAPHS

as applied in

WASTEWATER TREATMENT FACILITIES
and in the
MONITORING OF EFFLUENT WASTEWATERS

Instructional Package Worksheet

National Training and Operational Technology Center
Municipal Operations and Training Division
Office of Water Program Operations
U. S. Environmental Protection Agency

B. Preparation of Calibration Graphs

1. This skill is required in order to determine Phosphorus, Total Kjeldahl and Nitrate-Nitrite Nitrogen using the Effluent Monitoring Procedures (EMPs) for this course.
2. The procedure was written in the EMP format using a traditional method of plotting interdependent variables on an arithmetical graph form.
3. In the following Instructional Package Worksheet (IPW) for the EMP, the lesson plan is for each student to prepare one graph. If you choose a different assignment, adjust quantities of materials accordingly.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Preparation of calibration graphs

UNIT OF INSTRUCTION:

LESSON NUMBER: 1 of 1

ESTIMATED TIME: 60 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: If absorbance or transmittance determinations are made as part of an analysis being done for the permit system, the learner must know how to prepare a calibration graph in order to arrive at a number value for the chemical constituent being determined.

ENTRY LEVEL BEHAVIOR: The learner must:
1. Understand the terms milligram (mg), microgram (μg) and liter (l).
2. Have successfully completed the EMP, "Use of a Spectrophotometer."

A. INSTRUCTIONAL OBJECTIVE:
1. Terminal Behavior: The learner will prepare a calibration graph and use it to determine the amount of chemical constituent in a wastewater effluent sample.
2. Conditions: The learner will have the use of the attached EMP, a ruler, a pencil, and a sheet containing analysis data.
3. Accepted Performance: The graph will be prepared to the satisfaction of the instructor.

B. INSTRUCTIONAL RESOURCES:
1. Available Media:
    a. Six unassembled slides.
2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (sequencing):
All steps below are done in the classroom.
1. Have the learners read to themselves the Analysis Objectives and the Brief Description of Analysis. Ask for questions.
2. Distribute pencils and rulers to the learners.
3. Outline the procedure for preparing the calibration graph, using the six unassembled slides.
4. Have the learners do the entire EMP. Use the example data on Page 2-18 of the EMP, or the data obtained from the methylene blue color standards (see the EMP on the Use of a Spectrophotometer). In the latter case, note that the numbering of the two axes would be different than if the data on Page 2-18 were used.

5. Check the calibration graphs for use of the entire mg/l on A axes, and for reasonable neatness.
6. Final questions/discussion.

D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS

1. For each student:

   One ruler, 12 inches long
   Pencil
   Eraser
   Graph paper (one piece for each calibration graph). There are many kinds of graph paper. In ordinary water pollution analyses, a simple type of graph paper is used. Figure 1 is an example of one type of simple graph paper. The main feature of simple graph paper is that it is divided into a certain number of large squares of equal size. (For example, one inch might be the length of one side of the large squares). These large squares are subdivided into a certain number of smaller squares of equal size. (For example, a one inch square might be subdivided into one hundred small squares).

2. Shared:

   None

E. IPW REAGENT REQUIREMENTS (minimum amounts per student)

None

EXAMPLE DATA SHEET

ABSORBANCE VALUES OF STANDARDS	CONCENTRATION OF STANDARDS IN mg/l
0.000	0.0
0.130	5.0
0.290	10.0
0.420	15.0
0.570	20.0
0.690	25.0

Twenty-five ml of the sample were diluted to 500 ml in a volumetric flask. The absorbance of the diluted sample was 0.310. What is the concentration of the undiluted sample in mg/l?

A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES

for the

DETERMINATION OF TOTAL PHOSPHORUS (as P) OR OF ORTHOPHOSPHATE (as P), SINGLE REAGENT METHOD

as applied in

WASTEWATER TREATMENT FACILITIES
and in the
MONITORING OF EFFLUENT WASTEWATERS

Instructional Package Worksheet

National Training and Operational Technology Center
Municipal Operations and Training Division
Office of Water Program Operations
U. S. Environmental Protection Agency

CH.PHOS.ipw.2b.6.77

C. Determination of Total Phosphorus (as P) or of Orthophosphate (as P), Single Reagent Method

1. The Federal Register (FR) issuance in Outline No. 1 of this Guide lists one manual method to determine Total Phosphorus (as P) or Orthophosphate (as P): ascorbic acid reduction. To determine Total Phosphorus, the sample is pretreated by persulfate digestion to convert non-orthophosphate phosphorus forms to orthophosphate.

2. The persulfate digestion treatment and the ascorbic acid reduction method have been written in the Effluent Monitoring Procedure (EMP) format entitled, "Determination of Total Phosphorus (as P) or of Orthophosphate (as P), Single Reagent Method."

3. The reference source for the EMP is 1974 EPA "Methods for Chemical Analysis of Water and Wastes," p. 249. Other FR sources for the procedures to determine Total Phosphorus (as P) or Orthophosphate (as P) are:

   a. 14th ed., APHA, "Standard Methods for the Examination of Water and Wastewater," pp. 476 and 481 or 481 only.

   b. Part 31, 1975 "Annual Book of ASTM Standards," p. 384.

   c. Book 5, Chap. A1, 1970, USGS "Survey Techniques of Water - Resources Inventory," p. 133 or 131.

   d. 12th ed., AOAC, "Official Methods of Analysis of the Association of Official Analytical Chemists," p. 621.

4. The procedures in this EMP can be used for two determinations. Total Phosphorus (as P) can be determined using both the digestion and reduction procedures. If only Orthophosphate (as P) is the required data, only the reduction procedure is used.

5. In the following Instructional Package Worksheet (IPW) for the EMP, the lesson plan is for the determination of Total Phosphorus (as P) in a wastewater treatment plant effluent sample.

6. Each student is to process one blank, one standard and one sample. The IPW equipment and reagent requirements are based on this assignment. If you choose a different assignment, adjust quantities accordingly.

7. OPTIONAL assignments from this EMP have been used to check student proficiency at prerequisite basic skills for this course, i.e., using an analytical balance and making dilutions of stock solutions. These exercises can be used between lesson 1 and lesson 2 and require 1½ hours laboratory time. Details are in the IPW, C. INSTRUCTIONAL APPROACH. The related equipment and reagent requirements are noted on the IPW listings.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Total Phosphorus, mg/liter P

UNIT OF INSTRUCTION: Summary of 5 Elements

ESTIMATED TIME: Three hundred ninety minutes exclusive of any break time. If instructor chooses to check weighing and diluting skills (See C note), estimated time is 480 minutes.

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The determination of total phosphorus is required by many NPDES permits.

ENTRY LEVEL BEHAVIOR: Learner must have mastered the skills in Course I Basic Laboratory Skills, or have equivalent experience. In addition, learner must know how to operate a pH meter, assemble and use a membrane filtration assembly, operate a Bausch and Lomb Spectronic 20 Spectrophotometer, and construct and use a calibration graph.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will prepare glassware for the test, prepare a blank, a calibration standard and a sample dilution, digest them, filter them, adjust their pH, add color reagent, determine their absorbance, construct a calibration graph and use it to convert the absorbance reading for the sample into mg/liter P.

2. Conditions: The learner will be provided with classroom instruction, the required equipment, a suitable sample, a copy of the EMP, a copy of the data sheet, a copy of the EMP calibration graph form, and a total working time of 240 minutes.

3. Accepted Performance: His techniques must be satisfactory according to an instructor's rating and his answers must be the same as an Instructor's answers calculated from the same data.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: 1974 EPA "Methods for Chemical Analysis of Water and Wastes," EMP "Determination of Total Phosphorus (as P) or of Orthophosphate (as P), Single Reagent Method," eight overhead projecturals: (1) Federal Register listing of acceptable procedures; (2) definitions of reportable phosphorus fractions, 1974 EPA Methods Manual, p. 251; (3) EMP procedural flow sheet, p. 6; (4) word equations for "Basis of Test" and "Digestion;" (5) overview of the four lessons divided according to procedures in each lesson; (6) the Example Laboratory Data Sheet - EMP, p. 49; (7) overview of "Transfers" to be made during lesson three (Procedures G, I and J); (8) the Calibration Graph, EMP, p.50, and a 15 minute slide-tape, XT-44, "Determination of Phosphorus."

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH:

The method presented in this Effluent Monitoring Procedure is according to the 1974 EPA Methods Manual. Of the several reportable phosphorus fractions given in the manual, this EMP covers only total phosphorus and orthophosphate. Of these two, only total phosphorus is given in its entirety, with the additional information for orthophosphate being given as alternate steps in the procedure. Since many orthophosphate samples will need to be filtered before determination, and hence reported as dissolved orthophosphate, this procedure is discussed in the Training Guide, Section VII, E.3d.

The 1974 EPA Methods Manual specifies either a filter photometer or a spectrophotometer for use at either 650 or 880 nm. The steps in the procedure involving use of a spectrophotometer have been written specifically for use in conjunction with a B & L Spectronic 20 (or equivalent). If a filter photometer, uv-visible spectrophotometer or other machine is used, these steps will need to be modified. Even if a B & L Spectronic 20 (or equivalent) is being used, the standard instrument is not sufficient. Both the 650 and 880 nm wavelengths require that the standard phototube be replaced with an accessory infrared sensitive phototube and an accompanying filter.

The strength of the standard phosphorus solution, as given in the EMP, is predicated on the use of standard 1/2 inch colorimeter tubes in the spectrophotometer. As the 1974 EPA Methods Manual only specifies that the light path of the spectrophotometer be "1 cm or longer," Section VI,C.1b of the Training Guide gives a discussion of how to modify the standard phosphorus solution for cells with path lengths other than 1/2 inch, and presents an alternative Table 1 for use with 1 inch cells.

NOTE: Some instructors have utilized parts of this EMP to check student proficiency at the basic skills required to make reagents, i.e., using an analytical balance and making dilutions of stock solutions. EMP step B.12.6 can be used to check weighing technique. The instructor checks readings on the balance as the student obtains them. If volumetric glassware is available, the student can complete Procedure B.12 to make his own stock phosphorus solution. Alternatively, the instructor can prepare the stock solution for the entire class. Then, to check dilution technique, the students each do Procedure C. The evaluation standard is the calibration curve each obtains by using the standard Phosphorus Solution each prepared. For this to be valid, each student should prepare a calibration curve using only his own results (as opposed to using several absorbances obtained by other students). Assigning at least two calibration standards instead of one makes this feasible. These student exercises have been used between lesson 1 and lesson 2 and require 1 1/2 hours laboratory time.

1. Preparation for Instruction:

   a. Duplicate copies of the EMP data sheet and calibration graph form for each student.
   b. Have on hand a treatment plant effluent of known mg/liter P concentration so that a suitable dilution will be assigned. (A maximum of 50 ml per student will be needed.) Plan the assignment.

c. Make out tags for the sample bottles (1 per 3-4 students) including the information required on the EMP "Example Laboratory Data Sheet."
d. Use the EMP Training Guide VII directions to prepare phosphorus-free filter papers unless such filters were purchased.
e. See that equipment and reagents required for each student to do the planned "Student Performances" are assembled in the laboratory. (Hot 1:1 HCl must be available at the beginning of the first lab session.)

2. Sequencing:

a. Lesson one - 135 minutes
b. Optional - 90 minutes, checking basic skills: use of an analytical balance (B.12.6) and diluting solutions (C)
c. Lesson two - 60 minutes (This lesson does not have to immediately follow lesson one or the optional lesson.)
d. A break can be scheduled now before beginning lesson three, provided someone is available to watch the boiling digestions and remove them from the hot plates to cool at the end of the boiling period. However, no break is necessary as regards teaching the procedure.
e. Lesson three - 120 minutes (If necessary a break can be scheduled after G, filtration and pH adjustment, before beginning colorimetry.)
f. Lesson four - 30 minutes (This lesson on constructing and using the calibration curve to find mg/liter P in the sample does not have to immediately follow lesson three.)
g. Lesson five - 45 minutes (This wrap-up session may be scheduled anytime after the completion of the first four lessons.)

D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS

(1 reagent blank, 1 calibration standard and 1 sample dilution)

1. For each student: *

1 apron
12 boiling beads, glass, 4 per assignment
1 beaker, 400-600 ml for cleaning glassware
1 bottle, squeeze type for distilled water
1 bulb, pipet
1 filter assembly, smallest volume available to filter 10 ml digestate (filtrate gets transferred) Fritted glass funnels not recommended
3 pieces filter paper, phosphorus-free, 0.45 micron pore size, diameter to fit filter assembly (Gelman GA6 or equivalent)
3 flasks, 125 ml Erlenmeyer with wide mouth
3 flasks, 50 ml volumetric with stoppers (Need 4 if diluting sample)
1 pair forceps
1 funnel, to fit neck of 50 ml volumetric flask
1 pair safety glasses
1 pair rubber gloves for cleaning glassware
1 pH meter, preferably with combination electrode (If students share pH meters, teaching time becomes prohibitively long.)
1 pipet, Pasteur or Mohr for 10 N sodium hydroxide during pH adjustment
1 pipet, Mohr, 10 ml for combined reagent
1 pipet, 1 ml graduated in 0.1 ml for pH adjustments
1 pipet, volumetric, X ml or a graduate if diluting sample
1 pair tongs

* See NOTE, next page, bottom

1 copy data sheet in EMP
1 copy calibration graph form in EMP
If checking use of an analytical balance:
1 flask, 1 liter volumetric with stopper (if available)
1 spatula
1 weighing boat
If checking dilution technique:
1 flask, 1 liter volumetric with stopper
1 pipet, 20 ml volumetric

2. Shared: *

1-2 hoods (for cleaning glassware)
XXX hot plates to accommodate 3-125 ml Erlenmeyer flasks for each student
XXX pipets, volumetric, 1, 3, 5, 10, 20, 30, 40 and 50 ml (Numbers of each size needed depend on planned assignment of standards.)
XXX pipets, volumetric, 50 ml or volumes corresponding to planned assignment of sample dilutions
1 reagent spoon, 0.4 g, per 3 students for measuring ammonium persulfate
1 spectrophotometer for every 3 students
1 sample of known mg/liter P concentration split up into smaller containers (tagged), one for every 3 or 4 students
If checking use of an analytical balance:
1 analytical balance per 3 students

E. IPW REAGENT REQUIREMENTS: *

(minimum amounts per student)

2.5 liters phosphorus-free distilled water
150 ml warm 1:1 hydrochloric acid for use in cleaning glassware
150 ml combined reagent for test and for cleaning glassware
1.2 g ammonium persulfate
50 ml (maximum) standard phosphorus solution if students do not prepare their own
5 ml 10 N sodium hydroxide
5 ml 0.1 N sodium hydroxide
4 ml 11 N sulfuric acid (strong acid solution)
1 ml 1.1 N sulfuric acid
If checking use of an analytical balance:
0.2197 g potassium dihydrogen phosphate which has been dried for an hour at 105°C, then cooled and stored in a desiccator
If checking dilution technique:
about 30 ml stock phosphorus solution if students do not prepare their own stock

*NOTE: All glassware used for phosphorus tests and for preparation of phosphorus reagents must be acid-washed with hot 1:1 HCl, rinsed with distilled water and checked with combined reagent to assure complete removal of phosphorus contamination. If glassware can be reserved for phosphorus tests, this treatment is only required occasionally. See EMP Procedure A for details.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Total Phosphorus, mg/liter P

UNIT OF INSTRUCTION: Overview of the Determination and Cleaning Glassware (EMP Procedure A)

LESSON NUMBER: 1 of 5

ESTIMATED TIME: 135 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: To determine total phosphorus accurately in a sample, the learner must clean all glassware with hot acid.

ENTRY LEVEL BEHAVIOR: Learner must have mastered the skills in Course I, Basic Laboratory Skills, or have equivalent experience

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will clean his glassware according to EMP Procedure A.
2. Conditions: Learner will be given the equipment, a copy of the EMP, no assistance and 90 minutes.
3. Accepted Performance: Learner must accomplish terminal behavior to the satisfaction of an instructor rating. (Particularly note safety technique in handling the HCl acid.)

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of EMP; five overhead projecturals: Federal Register listing of acceptable procedures; definitions of reportable phosphorus fractions, 1974 EPA Methods Manual, p. 251; procedural flow sheet, EMP p. 6; word equations for "Basis of Test" and "Digestion;" overview of the four lessons; and a 15 minute slide-tape XT-44, "Determination of Phosphorus."
2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: (45 minutes)
   a. Lecture-Introduction
      -15 minute slide-tape presentation, XT-44, "Determination of Phosphorus"
      -Overheads - Federal Register, definition of reportable phosphorus fractions--discuss types of phosphorus (ortho vs. condensed and organic, etc.)
   b. Lecture - overheads of EMP flow sheet, "Basis of Test" and "Digestion" word equations, overview of four lessons. Then specifics of Procedure A.

c. Demonstration - cleaning of glassware, warning about dangers of hot HCl acid, and warning against use of phosphate detergents
d. Assignment - Have students read EMP Procedure A
e. Question and answer period on material presented
f. Assignment - Instructor should tell students items of glassware to be cleaned according to Procedure A. (Possibly a 1 liter flask, a volumetric pipet, and an Erlenmeyer flask).

2. Student Performance and Evaluation: (90 minutes)

As stated in Instructional Objective above, by instructor rating.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT: Total Phosphorus, mg/liter P

UNIT OF INSTRUCTION: Preparation of Phosphorus Calibration Standards and of Sample; Digestion of Standards, Blank, Sample (EMP Procedures D, E, and F)

LESSON NUMBER: 2 of 5

ESTIMATED TIME: 60 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: These procedures are part of the determination of total phosphorus in a sample.

ENTRY LEVEL BEHAVIOR: Learner must have mastered the skills in Course I, Basic Laboratory Skills, or have equivalent experience.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will prepare a blank, a calibration standard and a sample dilution according to EMP Procedures D and E, then digest these solutions according to EMP Procedure F, all as stated or described in the information column.

2. Conditions: Learner will be given the equipment, a copy of the EMP, a data sheet, no assistance and 45 minutes.

3. Accepted Performance: Learner must accomplish terminal behavior to the satisfaction of an instructor rating. (Particularly note correct pipetting technique and dilution to the exact 50 ml mark.)

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of EMP; overhead projecturals of flow sheet, EMP p. 6, overview of the four lessons and the data sheet, EMP p. 49.

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation (15 minutes)

   a. Lecture - overhead of EMP flow sheet: Relate Procedures D, E and F to the determination. Overhead of overview of the 4 lessons.
   b. Assignment - D. 1 Note division of establishing or checking curve. Assign blank plus one standard.
   c. Distribution - Give each student a copy of the data sheet.
   d. Overhead of Data Sheet--note keys to divisions
   e. Assignment - Instructor gives students sample information for E.1 and assigns to each a sample dilution.

NOTE: Once student solutions are all digesting, a logical continuity could be maintained by giving the students a break, then reassembling in the classroom for the next lesson. A staff member can watch the digestion volumes and remove flasks from the hot plates when digestion is complete.

2. Student Performance and Evaluation: (45 minutes)

As stated in Instructional Objective above, by instructor rating.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Total Phosphorus, mg/liter P

UNIT OF INSTRUCTION: Filtration and pH Adjustment of Digestions followed by Colorimetry and Spectrophotometric Measurements (EMP Procedures G, H, I and J)

LESSON NUMBER: 3 of 5

ESTIMATED TIME: 120 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: This is part of the laboratory procedure to determine total phosphorus, mg/liter P

ENTRY LEVEL BEHAVIOR: Learner must have mastered the skills in Course I, Basic Laboratory Skills, or have equivalent experience. In addition, learner must know how to operate a pH meter, assemble and use a membrane filtration assembly, and operate a spectrophotometer.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will filter his sample, standard and blank, adjust their pH, dilute them back to 50.0 ml, then add combined color reagent and determine their absorbances, according to the steps in EMP Procedures G, H, I and J as stated or described in the Information column.

2. Conditions: Learner will be given the equipment, the EMP, no assistance and 90 minutes.

3. Accepted Performance: Learner will perform Procedures G, H, I and J to the satisfaction of an instructor rating. (Particularly note pH meter technique, accuracy of dilution, and correct technique in the use of the spectrophotometer.)

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of the EMP, overheads of overview of the four lessons and overview of "Transfers" to be made in these procedures. Variety of filtration assemblies.

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: Briefing (30 minutes)

   a. Use overhead of overview of four lessons
   b. Assignment - Have students read Procedure G.
   c. Discussion
      (1) G.1. Show variety of filtration assemblies. Review use.
      (2) G.7. Go through notes on pH adjustment.
      (3) G.8. Note reason for this acid addition.

d. Briefly note contents of Procedures H, I, and J.
e. Use overhead of "Transfers" to be made in these procedures to give overview and to stress the importance of careful technique for quantitative recovery.
f. Assignment - If more than one spectrophotometer is being used, assign students to a particular instrument, emphasizing non-interchangeability of data from one instrument to another.

2. Student Performance and Evaluation: (90 minutes)

As stated in Instructional Objective above, by instructor rating.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Total Phosphorus mg/liter P

UNIT OF INSTRUCTION: Construction of a Calibration Curve and Reading, Calculating and Reporting Data from the Calibration Curve (EMP Procedures K, M, N and O)

LESSON NUMBER: 4 of 5

ESTIMATED TIME: 30 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The learner will be responsible for interpreting the data of the test.

ENTRY LEVEL BEHAVIOR: Learner must be able to construct and use a calibration curve.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will construct a calibration plot and use it to convert the absorbance reading for his sample into a mg/liter P concentration reading according to EMP Procedures K, M, N and O.

2. Conditions: Learner will be given the EMP, a sheet of graph paper, the absorbance data for a set of calibration standards, his data sheet and 15 minutes.

3. Accepted Performance: Learner's calibration curve and obtained result must be the same as the instructor's calibration curve and sample result using the same data.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of EMP; overheads of the Example Data Sheet and the calibration curve form in EMP, pp. 49 and 50

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: Lecture (15 minutes)

   a. Lecture - Discussion of EMP Procedures K, M, N and O. Use EMP Procedures K and M, and the overheads of the Data Sheet and the calibration curve form from the EMP to illustrate correct plotting procedure and use of a calibration curve.
   b. Distribution - Give each student a piece of graph paper.

2. Student Performance: (15 minutes) As stated in Instructional Objective above, by instructor rating.
   NOTE: The group, or groups, will need to pool data to construct a curve. Instructor may wish to have each group post their calibration standard data on the board.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Total Phosphorus, mg/liter P

UNIT OF INSTRUCTION: Follow-up and Summary of Subject Matter

LESSON NUMBER: 5 of 5

ESTIMATED TIME: 45 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: Learner should have an opportunity to ask any questions about the procedure and also should receive some final comments on sample dilutions, using other than 1/2 inch colorimeter tubes, the procedure modifications required to run an orthophosphate determination and the method of checking the continued accuracy of the calibration curve.

ENTRY LEVEL BEHAVIOR: Learner will have completed the entire Total Phosphorus EMP

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will have any unresolved questions about the procedure answered and will particularly consider the criteria for choosing sample volumes and rejecting the calibration curve.

2. Conditions: Copies of EMP, data sheet and calibration graph will be available.

3. Accepted Performance: Active participation in the 30 minutes wrap-up session as judged by the instructor.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: EMP, calibration graph and data sheet

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: Lecture-Discussion (45 minutes)

   a. Discuss results of test as appropriate. Answer questions.
   b. Note EMP Training Guide Sections VII, II and VI on dilutions, use of dilutions and using other-than-standard 1/2 inch colorimeter tubes. Answer questions.
   c. Note EMP Training Guide Section VII, E.3d on procedure modifications required to run orthophosphate determinations. Answer questions.
   d. Assignment - Have students read EMP Procedure D, steps 7 through 15, and EMP Procedure L, "Checking the Calibration Curve." Discuss and answer questions.
   e. Note EMP Procedure P, Clean-up.
   f. Answer any remaining questions.

2. Student Evaluation: Satisfactory participation in the session as judged by instructor.

Preceding page blank

A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES

for the

DETERMINATION OF CHEMICAL OXYGEN DEMAND

as applied in

WASTEWATER TREATMENT FACILITIES
and in the
MONITORING OF EFFLUENT WASTEWATERS

INSTRUCTIONAL PACKAGE WORKSHEET

National Training and Operational Technology Center
Municipal Operations and Training Division
Office of Water Program Operations
U. S. Environmental Protection Agency

CH.oc.ipw.2c.6.77

PART II INSTRUCTIONAL PACKAGE WORKSHEETS

D. Determination of Chemical Oxygen Demand

1. The Federal Register (FR) issuance in Outline No. 1 of this Guide lists one method to determine Chemical Oxygen Demand (COD): dichromate reflux.

2. The dichromate reflux method has been written in the Effluent Monitoring Procedure (EMP) format entitled, "Determination of Chemical Oxygen Demand."

3. The reference source for the EMP is 1974 EPA "Methods for Chemical Analysis of Water and Wastes," p. 20. Other FR sources for the procedure are:

   a. 14th ed., APHA, "Standard Methods for the Examination of Water and Wastewater," p. 550;

   b. Part 31, 1975, "Annual Book of ASTM Standards," p. 472;

   c. Book 5, chap. A 1, 1970, USGS, "Survey Techniques of Water - Resources Inventory," p. 124;

   d. 12th ed., AOAC, "Official Methods of Analysis of the Association of Official Analytical Chemists," p. 610;

   e. 1975, ANSI, "American National Standard on Photographic Processing Effluents," p. 17.

4. The procedures in this EMP are for low level COD of 5-50 mg/l but there are references to the Training Guide for the modifications required for greater concentrations.

5. In the following Instructional Package Worksheet (IPW) for the EMP, the lesson plan is for the determination of low level COD in a wastewater treatment plant effluent sample.

6. Each student is to process either one sample or one blank, and to standardize his ferrous ammonium sulfate titrant. The IPW equipment and reagent requirements are based on this assignment. If you choose a different assignment, adjust quantities accordingly.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Chemical Oxygen Demand

UNIT OF INSTRUCTION: Summary of 6 Elements

ESTIMATED TIME: 240 minutes excluding up to 70 additional minutes for a 2 hour oxidation period.

JUSTIFICATION: The reporting of Chemical Oxygen Demand is required by many National Pollutant Discharge Elimination System permits.

ENTRY LEVEL BEHAVIOR: The learner must know how to use volumetric glassware, pipet bulbs and burets; how to perform titrations; and how to subtract, multiply and divide numbers containing decimals.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior - The learner will record sample identification information, prepare one sample or one blank for oxidation, standardize ferrous ammonium sulfate solution and, after rinsing and removing his flasks from the condensers, use the FAS for titration of the sample or blank. He will clean up his equipment, then use his laboratory data to calculate the chemical oxygen demand of his sample.

2. Conditions - He will be given classroom instruction, the required equipment conditioned and ready to use, the required reagents, a sample, a copy of the related EMP, copies of each of the data sheets in the EMP and total working time of 160 minutes.

3. Accepted Performance - His techniques must conform to those presented in the EMP according to an Instructor's rating. His volume for the ferrous ammonium sulfate standardization should be within ±0.05 ml of a majority of the class results and his COD value should be within ±4mg/liter of the class average unless some explanation of disagreement is accepted by the Instructor.

B. INSTRUCTIONAL RESOURCES:

1. Available Media - EPA "Methods for Chemical Analysis of Water and Wastes," 1974; EMP "Determination of Chemical Oxygen Demand;" copies of the EMP Data Sheets; two referenced papers: Dean, Williams, Wise: "Disposal of Mercury Wastes from Water Laboratories," Environmental Science and Technology, Vol. 5, No. 10, 1971, p. 1044; Maag and Hecker: "Recovery of Mercury in Solution," Journal of Environmental Quality, Vol. 1, No. 2, 1972, p. 192; copies of two data sheets from the EMP; 34 assembled slides, numbered according to their use with the lessons in this IPW; 3 overheads of EMP pages on "Flow Sheet" and two data sheets plus 1 overhead on rules for rounding off numbers.

2. Suggested Media - none

C. INSTRUCTIONAL APPROACH:

1. Preparation for Instruction:
   - a. Duplicate copies for each student of the two EMP data sheets. Two completed copies of each will be required. You can distribute one and later duplicate the completed sheet or you can distribute two and have the student make the copy.
   - b. Duplicate for each student a copy of the two papers about mercury disposal.
   - c. Obtain a sample (75 ml per student) of treatment plant effluent with COD between 5-50 mg/liter.
   - d. Make out tags for the sample bottles (1 per 3 students) including the information required on the EMP "Typical Laboratory Data Sheet."
   - e. See that the equipment and reagents required for each student to do the planned "Student Performances" are assembled in the laboratory.

2. Sequencing:
   - a. Lesson one - 100 minutes.
   - b. A break can be scheduled next, of any time duration provided someone is available to turn off the reflux apparatus two hours after lesson one ends. However, no break is necessary as regards teaching the procedure.
   - c. Lesson two - 50 minutes.
     This standardization procedure should be scheduled on the same day as lesson three if possible.
   - d. Lesson three - 45 minutes.
   - e. Lesson four - 30 minutes.
     This lesson on calculations does not have to immediately follow lesson three.
   - f. Lesson five - 20 minutes.
     This discussion of variable aspects of the test does not have to immediately follow lesson four.
   - g. Lesson six - 10 minutes.
     This discussion of the corrected data sheets will have to be scheduled after the Instructor collects (lesson 4) and corrects (outside class time) the data sheets.

D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS
(1 sample or blank, 1 FAS standardization titration)

1. For each student: *
   - 1 Apron
   - 1 Beaker, glass, 150 ml - 250 ml (to transfer FAS)
   - 1 Buret, 25 ml, 0.1 ml graduations, teflon stopcock
   - 1 Clamp, buret, for titration stand
   - 1 Cylinder, graduated, 25 ml
   - 1 Cylinder, graduated, 100 ml
   - 1 Evaporating dish (to place between flask and heating surface for cooling)

* See NOTE, next page, bottom

1. For each student (continued) *
   - 1 Flask - condenser - heating surface assembly with tubing connections to a source of cooling water.
   - 5 Beads, glass boiling, in the reflux flask
   - 1 Flask, Erlenmeyer, wide mouth 500 ml, with volumes marked on the outside.
   - 1 Funnel, short stem (to fill 25 ml buret)
   - 1 Pair safety glasses
   - 1 Pan for ice water to cool mixture in reflux flask at various stages of the test.
   - 1 Pencil, wax
   - 1 Pipet bulb, preferably propipet to avoid accidents when pipeting H_2SO_4
   - 1 Pipet, graduated, 10 ml
   - 1 Pipet, volumetric, 10 ml.
   - 1 Pipet, volumetric, 25 ml.
   - 1 Pipet, volumetric, 50 ml.
   - 1 Stand, titration, support for buret.
   - Towels, paper
   - 1 Wash bottle, squeeze type 500 ml

* NOTE: All glassware should be conditioned for use in the test according to EMP Procedures D and E.

2. Shared:
   - 1 Two liter container per 6 students for test wastes containing mercury complexes and significant amounts of sulfuric acid.
   - 1 Buchner funnel for each test-wastes container (to catch glass beads from flask)
   - 1 Reagent spoon per 3 students to roughly measure 1 gram of mercuric sulfate.
   - 1 Sample bottle, tagged, per 3 students. Should preferably be glass but plastic may be used if it is known that no organic contaminants are present.

E. IPW REAGENT REQUIREMENTS: (minimum amounts per student)

175 ml distilled water, high quality, low COD
11 drops ferroin
75 ml 0.025 N ferrous ammonium sulfate solution (dilute 100.0 ml of 0.250 N per 1 liter)
1 g mercuric sulfate
50 ml 0.025 N potassium dichromate solution (dilute 100.0 ml of 0.250 N per 1 liter)
40 ml concentrated sulfuric acid
85 ml sulfuric acid-silver sulfate solution

NOTE: Student will need a tray of ice cubes for cooling bath during test.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Chemical Oxygen Demand

UNIT OF INSTRUCTION: Procedure A Preparing to Test the Sample and Procedure F Oxidation of the Sample and Blank

LESSON: 1 of 6

ESTIMATED TIME: 100 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The learner must perform these procedures to determine COD.

ENTRY LEVEL BEHAVIOR: The learner must know how to use volumetric glassware and pipet bulbs.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior - The learner will record sample identification information, prepare one sample or one blank in a reflux flask, then attach the flask to a condenser and begin heating it, all according to EMP Procedures A and F.

2. Conditions - He will be given classroom instruction, a copy of the EMP and a related data sheet, the required reagents (Procedure B), the required equipment conditioned and ready to use (Procedures D and E), a sample, minimal supervision and total working time of 40 minutes.

3. Accepted Performance - His techniques must conform to those described in the EMP according to an Instructor's rating. (Particularly note caution in pipeting/measuring sulfuric acid and transferring it to the flask).

B. INSTRUCTIONAL RESOURCES:

1. Available Media - EMP; 19 slides pertaining to overview of test (2), Procedure A. Step 2 Reagents (2), A. Step 3 Conditioning (3), A. Step 4 Conditioning (1) and Procedure F (11); 1 overhead and copies of the EMP "Typical Laboratory Data Sheet."

2. Suggested Media - None

C. INSTRUCTIONAL APPROACH:

1. Presentation (60 minutes)
   a. Introduction - Training Guide I, paragraph 1.
   b. Lecture using board - EMP p.4-4 - Reaction, then Quantitation
      1) Develop equation with essentials first: reagents - oxidation time - temperature.
      2) Equipment: function of condenser
      3) Catalyst: define "catalyst" - Ag_2SO_4 - why.
      4) Chloride interference - effect on catalyst - effect on potassium dichromate - adding $HgSO_4$.
      5) Want excess $K_2Cr_2O_7$ to titrate and then calculate COD.
      6) Blank - why necessary - used in calculations.

Preceding page blank

c. Use 19 slides to teach overview of test (2); Procedure A and its references to Procedures B(2), D(3) and E(1); Procedure F(11)

d. Go through A steps 1-4 to note references and locations of EMP Procedures B, D, E.

e. A Step 5 - Distribute copies of EMP "Typical Laboratory Data Sheet." (Option: Two completed sheets will be required for each student. You can distribute 1 and later duplicate the completed sheet or you can distribute 2 and have the student make the copy).

f. Note left column on data sheet contains step designation, the right column contains example numbers. (Overhead can be used)

g. Have students make a note at the end of A Step 5 to turn to Procedure F for the next part of laboratory assignments.

h. Procedure F is written to do 1 sample and 1 blank.
   1) Assign the blank to 2 students, warning them to read carefully and just do F steps pertaining to a blank.
   2) Rest of class are to do steps pertaining to a sample and omit steps referring to the blank.
   3) F Step 12 - Stress hazards of H_2SO_4. Demonstrate using a pipet bulb or an automatic dispenser.

2. Student Assignment - Do Procedure A step 5 and all of Procedure F in the laboratory

3. Student Performance and Evaluation (40 minutes)
   As stated in the Instructional Objective above.

4. NOTE: It is not essential that the mixtures boil exactly 2 hours for this exercise. This IPW provides 50 minutes of other instruction before the heat sources must be cut off.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Chemical Oxygen Demand

UNIT OF INSTRUCTION: Procedure C. Standardizing Ferrous Ammonium Sulfate Solution

LESSON: 2 of 6

ESTIMATED TIME: 50 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: On the day of use, the learner must standardize the ferrous ammonium sulfate solution.

ENTRY LEVEL BEHAVIOR: The learner must know how to use volumetric pipets, pipet bulbs, burets and how to titrate a solution to a color change end point.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior - The learner will titrate one aliquot of the ferrous ammonium sulfate solution, record his result, use the posted results of the class as "duplicate" results to check his work, and then calculate the normality of the solution, all according to EMP Procedure C.

2. Conditions - He will be given classroom instruction, a copy of the EMP and a related data sheet, the required reagents (Procedure B), the required equipment conditioned and ready to use (Procedure D and E), minimal supervision and total working time of 40 minutes.

3. Accepted Performance - His techniques must conform to those described in the EMP according to an Instructor's rating. (Particularly note the drop-by-drop addition technique near the end point of the reaction). His total ml added should be equal to or within ±0.05 ml of a majority of the class results as determined by the Instructor who corrects the completed data sheets.

B. INSTRUCTIONAL RESOURCES:

1. Available Media - EMP; 1 overhead of EMP Flow Sheet; copies of the EMP data sheet for "Standardization of Ferrous Ammonium Sulfate Solution" and 1 overhead of same; 6 slides pertaining to overview of Procedure C (4) and the color change at the end of the titration (2); 1 overhead reviewing rules for rounding of numbers.

2. Suggested Media - None

C. INSTRUCTIONAL APPROACH

1. Presentation (10 minutes)
   a. Use overhead of Flow Sheet so students see where they are in doing the test.
   b. Use 4 slides to give overview of Procedure C.
   c. Distribute copies of EMP data sheet for "Standardization of Ferrous Ammonium Sulfate Solution." (Option: Two completed data sheets will be required for each student. You can distribute 1 and later duplicate the completed sheet or you can distribute 2 and have the student make the copy).

d. Use overhead to show left column on data sheet contains step designation, the right column contains examples for duplicate titrations.

e. The EMP has directions to do duplicate titrations. For this class exercise each student should do one titration and use class results as a check according to Procedure C. Thus all students should omit steps beginning with the word "Repeat" (Steps 3, 10, 15, 32).

f. Present 2 slides which show the color change for the end point of the titration.

2. Student Assignments - Do Procedure C through step 33 then turn off the heat under your test flask and insert an evaporating dish upside down between the flask and heating surface. Go to the classroom and finish Procedure C.

3. Discussion of Duplicates - When all the students are back in the classroom, put their ml results from C. step 33 on the board and go through C. step 34 on allowable differences for duplicates.

4. Review of Rounding Off Numbers - Use overhead to review for C. step 37 rounding off operation which is recorded as C. 38 on the data sheet.

5. Student Performance and Evaluation (40 minutes)
As stated in the Instructional Objective above.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Chemical Oxygen Demand

UNIT OF INSTRUCTION: Procedure G. Rinsing and Removing Flasks from Condensers, Procedure H. Quantification: Titration of Sample and Blank, and Procedure I. Clean Up.

LESSON: 3 of 6

ESTIMATED TIME: 45 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The learner must perform these procedures to determine Chemical Oxygen Demand.

ENTRY LEVEL BEHAVIOR: The learner must know how to use a buret and how to titrate a solution to a color change end point.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior - The learner will rinse and remove his reaction flask from the condenser, titrate the excess potassium dichromate in it with the ferrous ammonium sulfate solution he standardized in lesson 2, record his results, and clean up his equipment, all according to EMP Procedures G, H and I.

2. Conditions - He will be given classroom instruction, a copy of the EMP and a related data sheet, the required reagents (Procedure B), the required equipment conditioned and ready to use (Procedures D and E), minimal supervision and total working time of 30 minutes.

3. Accepted Performance - His techniques must conform to those described in the EMP according to an Instructor's rating. (Particularly note the drop-by-drop technique near the end point of the reaction).

B. INSTRUCTIONAL RESOURCES:

1. Available Media - EMP; copies of the EMP Data Sheet; 1 overhead of EMP Flow Sheet; slides pertaining to Procedure H titration color change (use the 2 from lesson 2) and 3 slides of rinsing and removing the flasks from the condensers and bringing the solution to volume.

2. Suggested Media - None

C. INSTRUCTIONAL APPROACH:

1. Presentation (15 minutes)
   a. Use overhead of Flow Sheet so students see where they are in doing the test.
   b. Re-project the 2 slides of the color change at the end point of titration in Procedure C to stress that Procedure H is the same process.
   c. Contrast and compare Procedure H to Procedure C.
      1) Only add ferroin to the diluted test mixture, then titrate.
      2) Add 10 drops of ferroin this time since the diluted volume is greater here.

   d. Use 3 slides to preview Procedure G, removing flasks.
   e. Procedures G and H are written for testing a sample and a blank. Use the steps that apply to your assignment. (See next section).

2. Student Assignment - Do Procedures G, H, and I in the laboratory. For G, all do steps 1 through 10; for H, all do steps 1 through 16; for I, all do all steps.

3. Student Performance and Evaluation (30 minutes)
   As stated in Instructional Objective above.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Chemical Oxygen Demand

UNIT OF INSTRUCTION: Procedure J. Calculations

LESSON: 4 of 6

ESTIMATED TIME: 30 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The learner may be responsible for using the test data to calculate the mg/liter Chemical Oxygen Demand.

ENTRY LEVEL BEHAVIOR: The learner must know how to subtract, multiply, and divide numbers containing decimals and how to round off numbers.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior - The learner will use his laboratory result to calculate the Chemical Oxygen Demand of his sample according to EMP Procedure J and report that result to the Instructor. He will also turn in his completed data sheets for the sample and for standardizing the ferrous ammonium sulfate solution.

2. Conditions - He will have the EMP and the related data sheets he used during earlier lessons, the results for titrating the blank, supervision and a total working time of 20 minutes.

3. Accepted Performance - The data sheets must be complete and with the same final answer as the Instructor calculates from the same data. Also, his value for COD must agree within ±4 mg/liter of the class average unless some explanation of disagreement is accepted by the Instructor.

B. INSTRUCTIONAL RESOURCES:

1. Available Media - EMP, copies of EMP Data Sheets, overheads (3) of the EMP Flow Sheet, the EMP Data Sheet and examples of rounding off numbers.

2. Suggested Media - None

C. INSTRUCTIONAL APPROACH:

1. Introduction (1 minute)
   a. Use overhead of Flow Sheet to show students where they are in the EMP.
   b. Procedure J has step-by-step directions on using the data sheet to calculate COD. The steps follow from J.1a, the calculation formula for COD which is also printed at the bottom of the data sheet. Use overhead to show area of data sheet used for calculations.

2. Presentation (4 minutes)
   a. Note A = ml FAS to titrate the blank. Post the ml values from the 2 students who did blanks.
   b. Discuss the criteria for acceptable blanks in Training Guide VII.
   c. If both blank volumes are acceptable, average the numbers.
   d. If only one blank volume is acceptable, use just that one.
   e. Have the class record the blank value on their data sheets in the column they used for their sample, on line 9.

3. Student Assignment - Use the steps in Procedure J and your own results on your data sheet to calculate the COD of your sample. The two students who did the blank should also do the calculation, using 50.0 ml on line 6 and 10.0 ml on line 10. During this time some may need to see the overhead on rounding off numbers.

4. Discussion (5 minutes)
   As students get results, see that they are posted on the board. When all are available, discuss them and give the students a chance to deduce causes for any far-ranging numbers. Some points to be covered are novice technique, differing FAS normality values, the effect of the 8000 factor.

5. Collect completed data sheets from the students.

6. Student Performance (20 minutes)
   As stated in the Instructional Objective above.

7. Student Evaluation
   The Instructor will correct the data sheets outside of class time. On the standardization sheet, accepted performance is a result agreeing ±0.05 ml with a majority of the class results. On the laboratory data sheet, accepted performance is a result agreeing ±4mg/liter with the class average unless some explanation of disagreement is accepted by the Instructor. A corrected copy of each sheet is to be returned to the student and a second corrected copy of each sheet is to be retained by the Instructor for the course records.

   Students often "slip up" on the following items on the data sheet:
   a. They fail to record "Name of Plant."
   b. Lines 3 and 5 each require two pieces of information. They often record only one.

   Correcting the sheets gives an opportunity to check:
   a. If student reads and records buret volumes to two decimal places (lines 7 through 11).
   b. If student correctly rounds off numbers (line 12 is result from FAS Sheet; line 16).

   A detail that can also be noted is using a zero with decimal points as in line 6 (50.0) and line 12 (0.02X).

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Chemical Oxygen Demand

UNIT OF INSTRUCTION: Final Comments on Training Guide Topics - Smaller Volumes of Sample (VII), Automatic Dispensers (V), Two Hour Oxidation Period (VII), and Disposal of Mercury - Containing Wastes (VI).

LESSON: 5 of 6

ESTIMATED TIME: 20 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: In the future the learner may have to make decisions to modify these aspects of the procedure and he should learn the basis for making these choices.

ENTRY LEVEL BEHAVIOR: Learner should have completed lessons 1 through 4.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior - The learner will particularly consider the topics for this unit of instruction and have any unresolved questions about this information answered.

2. Conditions - He will have his EMP, copies of two papers on mercury disposal, and an opportunity for asking questions during the 20 minute session.

3. Accepted Performance - Active participation in the session as judged by the Instructor.

B. INSTRUCTIONAL RESOURCES:

1. Available Media - EMP and copies of two papers on mercury disposal - Dean, Williams, Wise: "Disposal of Mercury Wastes from Water Laboratories," Environmental Science and Technology, Vol. 1, No. 10, 1971, p. 1044 and Maag and Hecker: "Recovery of Mercury in Solution," Journal of Environmental Quality, Vol, 1, No. 2, 1972, p. 192. Also 6 slides pertaining to Training Guide Topics.

2. Suggested Media - None

C. INSTRUCTIONAL APPROACH:

1. Introduction (2 minutes)
   Go through the use of keys in the fourth column of the EMP to locate related information in the Training Guide.

2. Discuss the Unit Topics from the Training Guide (18 minutes)
   a. Smaller Volumes of Sample for the Test (VII) - 1 slide
      1) Especially note point 6 regarding volumes.
   b. Automatic Dispensers (V) - 1 slide
      1) Especially for acids
      2) Last two paragraphs have amounts
      3) You must check delivered volumes.

c. Two Hour Oxidation (Boiling) Period (VII) - 1 slide
    1) Must do 7 duplicates over a period of several days so variability of the sample source is more likely to show up.
    2) Results must agree within the precision limits of the test, ±4 mg/liter.
    3) Should re-check periodically by running duplicates.

d. Disposal of Mercury-Containing Wastes (VI) - 3 slides
    1) Pass out copies of the two papers cited in the Training Guide.
    2) Dean, et al: Raise the pH to 10 or higher and use a 40-50% w/v solution of sodium thiosulfate to precipitate the mercury as a sulfide. Note list of reprocessors.
    3) Maag and Hecker: Use aluminum (foil) to displace mercury which can then be recovered in metallic form.

3. Student Performance and Evaluation - As stated in Instructional Objective above.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Chemical Oxygen Demand

UNIT OF INSTRUCTION: Discussion of Corrected Data Sheets

LESSON: 6 of 6

ESTIMATED TIME: 10 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The learner should have an opportunity to question any corrections made on his data sheet.

ENTRY LEVEL BEHAVIOR: Learner should have completed lessons 2 and 4.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior - The learner will ask any questions or give any explanations regarding his corrected data sheets.

2. Conditions - He will have EMP and corrected data sheets, and 10 minutes.

3. Accepted Performance - Active participation in the session as judged by the Instructor.

B. INSTRUCTIONAL RESOURCES:

1. Available Media - EMP, corrected data sheets.

C. INSTRUCTIONAL APPROACH:

1. Discussion of Corrected Data Sheets (10 minutes)
   a. Distribute corrected data sheets for the sample and for standardizing the ferrous ammonium sulfate solution.
   b. Ask if there are any questions or explanations about notes made on any of the sheets.
   c. Make any observations that are of general interest to the class or which stress critical aspects of the test.

2. Student Performance and Evaluation - As stated in the Instructional Objective above.

A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES

for the

DETERMINATION OF TOTAL KJELDAHL NITROGEN

as applied in

WASTEWATER TREATMENT FACILITIES
and in the
MONITORING OF EFFLUENT WASTEWATERS

INSTRUCTIONAL PACKAGE WORKSHEET

National Training and Operational Technology Center
Municipal Operations and Training Division
Office of Water Program Operations
U.S. Environmental Protection Agency

CH.N.IPW.1.6.77

E. Determination of Total Kjeldahl Nitrogen

1. The Federal Register (FR) issuance in Outline No. 1 of this Guide lists one method for the first phases of the Total Kjeldahl Nitrogen Test: digestion of the sample to convert certain nitrogen forms to ammonia and distillation to isolate the ammonia for quantification. It lists three manual methods for the subsequent measurement of the ammonia: nesslerization (for concentrations of 0.05-1 mg/l), titration (for 1-25 mg/l) and electrode (for concentrations from 0.05-1400 mg/l).

2. The digestion, distillation, nesslerization and titration methods for macro or micro sample volumes have been written in the Effluent Monitoring Procedure (EMP) format entitled, "Determination of Total Kjeldahl Nitrogen." There is a separate EMP for the electrode method ("Determination of Ammonia by an Ammonia Selective Electrode") which can be used for the ammonia quantification part of the Total Kjeldahl Nitrogen Determination.

   To determine only Organic Nitrogen, a sample is processed for Total Kjeldahl Nitrogen, and also for original Ammonia. Organic Nitrogen is the difference between these two. Organic Nitrogen can be determined directly by removal of ammonia from the sample (by boiling or distillation) prior to the digestion procedure of the Total Kjeldahl Nitrogen Test. In addition to the Selective Electrode EMP, there is an EMP for the distillation, nesslerization and titration methods for macro sample volumes, "Nitrogen, Ammonia Determination."

3. The reference source for the Total Kjeldahl Nitrogen EMP is 1974 EPA "Methods for Chemical Analysis of Water and Wastes," p. 175. Other FR sources for the procedure are:

   a. 14th ed., APHA, "Standard Methods for the Examination of Water and Wastewater," p. 437;
   b. Book 5, Chap. A1, 1970, USGS, "Survey Techniques of Water-Resources Inventory," p. 122;
   c. 12th ed. AOAC, "Official Methods of Analysis of the Association of Official Analytical Chemists," p. 612.

4. The procedures in this EMP provide a choice of macro or micro sample volumes, and a choice of nesslerization or titration for the Ammonia measurement.

5. In the following Instructional Package Worksheet the lesson plan is for digestion and distillation of a micro volume of a WWTP effluent sample followed by nesslerization for the ammonia quantification.

6. Each student is to process one sample through the digestion, distillation and nesslerization procedures and also one blank and two standards through the nesslerization procedure. The IPW equipment and reagent requirements are based on this assignment. If you choose a different assignment, adjust quantities accordingly.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Total Kjeldahl Nitrogen

UNIT OF INSTRUCTION: Summary of 3 elements

ESTIMATED TIME: 5 hours

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The reporting of Total Kjeldahl Nitrogen is required by many National Pollutant Discharge Elimination System permits.

ENTRY LEVEL BEHAVIOR: The learner must have completed Course I, Basic Laboratory Skills or have shown competence in the areas covered by this course. Course II, Basic Parameters for Municipal Effluents, is not a prerequisite, but is desirable. The learner must also be able to:

1. Adjust pH of a sample

2. Use a spectrophotometer

3. Plot a calibration curve

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will perform all laboratory work to determine Total Kjeldahl Nitrogen by the micro method and Nesslerization, then will calculate results.

2. Conditions: The learner will have available all necessary glassware, reagents and equipment, and a copy of the Total Kjeldahl Nitrogen Effluent Monitoring Procedure.

3. Accepted Performance: The learner will choose and properly deliver the correct reagents, correctly prepare a calibration curve and correctly perform the final calculations. He must obtain at least 80% on an overall evaluation of these tasks (checklist in Lesson 3), or else repeat the analysis until he does obtain the 80% rating.

B. INSTRUCTIONAL RESOURCES

1. Available Media

   a. Total Kjeldahl Nitrogen Effluent Monitoring Procedure (EMP)

   b. 1974 EPA Methods of Chemical Analysis, page 175.

   c. Transparencies available from National Training and Operational Technology Center (NTOTC), OT-9(N) Series of 9: the figures from Total Kjeldahl Nitrogen EMP.

   d. A Performance Checklist is part of lesson 3.

2. Suggested Media: - Video Tape presentation showing both micro and macro methods of the Total Kjeldahl Nitrogen Determination.

C. INSTRUCTIONAL APPROACH: The method presented in this Effluent Monitoring Procedure is according to the 1974 EPA Chemical Methods Manual.

The Total Kjeldahl Nitrogen Determination may be performed by using either the micro method or the macro method. The procedure has been written primarily for the micro method, although suggested procedures and equipment are listed in the Effluent Monitoring Procedure for the macro method. It should be noted that the micro method requires a micro Kjeldahl Digestion unit and a steam distillation apparatus. If this equipment is used then the determination may be performed in the 5-hour time frame. The macro method would require a macro digestion and distillation apparatus, and the time required would be 7-hours.

In order to perform the analysis in the 5-hour time frame, all reagents except for the colorimetric standards must be prepared by the Instructor. These are itemized later in this Instructional Package Worksheet.

1. Preparation for Instruction:

   a. Duplicate copies of the EMP Data Sheet and Calibration Graph form for each student.
   b. Have on hand a treatment plant effluent sample of known TKN concentration, so that a suitable dilution will be assigned. (a maximum of 50 milliliters per student will be needed.)
   c. Make out tags for the sample bottles. Include information required on the EMP example laboratory data sheet.
   d. Make sure that all equipment and reagents required for each student to do planned student performances are assembled in the laboratory. For the micro method with a maximum class size of 12, two digestion racks are needed and at least three separate steam distillation units should be available. This allows each student to have his/her own sample. Since the steam distillations only require 10 minutes per sample, three are sufficient.

      If the macro method is chosen for instruction, then a digestion rack with 12 burners is recommended as well as a distillation rack capable of holding the same number. For the other macro equipment and reagents, see Chapter 8 in this Staff Guide.

      The equipment needed for the Total Kjeldahl Nitrogen Determination is somewhat complex and consequently should be checked out thoroughly by the Instructional Staff, prior to course time.

2. Sequencing:

   a. Lesson one-60 minutes. Theoretical Concepts of the Nitrogen Determination to be discussed in the classroom.
   b. Lesson two-45 minutes. It is advantageous to explain only the digestion and to have the student set up and begin digestion of his sample. Then the Instructor can give the complete procedure description, including the utility of the micro and macro methods, sample handling and preservation and a demonstration of both micro and macro pieces of equipment.

c. A break can be scheduled, such as a lunch break, during the digestion of the sample.

d. Lesson three - 3½ hours. Analysis of the sample. This includes the completion of the digestion, steam distillation, colorimetric determination, calculations and discussion of results.

D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS: (1 sample for digestion, plus 1 blank and 2 standards for Nesslerization)

1. For each student:

a. 1 apron
b. 6 boiling beads
c. 1 50 ml beaker, graduated
d. 1 100 ml beaker, graduated
e. 2 150 ml beakers, graduated
f. 2 10 ml cylinders, graduated
g. 1 100 ml cylinder, graduated
h. 1 50 ml Erlenmeyer flask, graduated
i. 1 125 ml Erlenmeyer flask, graduated
j. 1 100 ml Kjeldahl flask
k. 4 Nessler tubes scored at 50 ml
l. 2 10 ml pipets (Mohr) graduated
m. 1 xx ml pipet, volumetric, of size required if sample dilution is necessary
n. 1 50 ml pipet, volumetric
o. 4 No. 3 or No. 6 rubber stoppers or caps to fit the Nessler tubes

2. Shared:

a. Digestion apparatus, micro, 1 for every 6 students
b. Distillation apparatus, steam, micro, 1 for every 4 students
c. Spectrophotometer, 1 for every 3 students
d. Nessler tube support, 1 for every 2 students

E. IPW REAGENT REQUIREMENTS:
(minimum amounts per student)

a. 5 ml boric acid solution, 2%
b. 10 ml sodium hydroxide-sodium thiosulfate solution
c. 2 drops phenolthalein solution
d. 4 ml Nessler reagent
e. 0.18 ml stock ammonium chloride solution
f. 10 ml digestion reagent

INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Total Kjeldahl Nitrogen

UNIT OF INSTRUCTION: Theoretical concepts

LESSON NUMBER: 1 of 3

ESTIMATED TIME: 1 hour

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The student should know what forms of nitrogen this test measures and the significance of the test in waste treatment plant operations.

ENTRY LEVEL BEHAVIOR: The learner must have completed Course I, Basic Laboratory Skills, or have shown competence in the areas covered by this course. Course II Basic Parameters, is not a prerequisite, but is desirable.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will participate in classroom discussion.
2. Conditions: The learner will have a copy of the Total Kjeldahl Nitrogen Effluent Monitoring Procedure.
3. Accepted Performance: The learner will answer all questions orally given by the instructor.

B. INSTRUCTIONAL RESOURCES:

1. Available Media:
   a. Effluent Monitoring Procedure Training Guide Notes, Section I
   b. Transparencies available from NTOTC, OT-9 (N) Series of 9. Use #8, Nitrogen Cycle and #9, Nitrogen Transformations.
2. Suggested Media:

C. INSTRUCTIONAL APPROACH: (Sequencing)

1. Presentation (45 minutes)

   Instructor should follow the outline in Training Guide, Section I of the Effluent Monitoring Procedure to introduce the student to the utility of the Total Kjeldahl Nitrogen Determination. The transparencies mentioned above should be used in conjunction with the lecture material.

2. Student Performance & Evaluation (15 minutes)

   As stated in the Instructional Objective above by Instructor rating.

Preceding page blank

INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Total Kjeldahl Nitrogen

UNIT OF INSTRUCTION: Procedure Description

ESTIMATED TIME: 45 minutes LESSON NUMBER: 2 of 3

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The student must be instructed in the procedure steps so he can perform the determination in the laboratory.

ENTRY LEVEL BEHAVIOR: The learner must have completed Course I, Basic Laboratory Skills or have shown competence in the areas covered by this course. Course II Basic Parameters, is not a prerequisite, but is desirable. He must have completed Lesson 1 of this IPW.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will begin digestion of his sample, then participate in classroom discussion of the complete determination.
2. Conditions: The learner will have a copy of the Effluent Monitoring Procedure, Total Kjeldahl Nitrogen Determination.
3. Accepted Performance: The learner will answer all questions orally given by the Instructor.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Effluent Monitoring Procedure, Total Kjeldahl Nitrogen and transparences available from NTOTC, OT-9 (N) Series of 9. Use:
   a. #1 Macro Digestion Apparatus
   b. #2 Macro Distillation Apparatus
   c. #3 Micro Digestion Apparatus
   d. #4 Micro Steam Distillation Apparatus
   e. #5 Micro Steam Distillation Apparatus
2. Suggested Media: Video Tape showing both micro and macro methods for TKN

C. INSTRUCTIONAL APPROACH: (Sequencing)

1. Presentation (30 minutes)
   a. Instructor should follow the outline of the procedures covering topics listed. He should explain the digestion step and have the students go to the laboratory to start digestion of a sample. Then the students return to the classroom and the Instructor discusses the complete procedure.

Preceding page blank

b. It should be emphasized again at this point that the Total Kjeldahl Nitrogen can be performed by either the macro or the micro method, and the differences should be noted. In addition, the difference between the colorimetric and titrimetric determinations should be explained. The flow chart on page 5-5 of the EMP should be used for this purpose.

2. Student Performance & Evaluation: (15 minutes)

As stated in Instructional Objective above by Instructor rating.

INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Total Kjeldahl Nitrogen

UNIT OF INSTRUCTION: Analysis

LESSON NUMBER: 3 of 3

ESTIMATED TIME: 3¼ hours

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The student should perform the determination with Instructor observation and supervision so he can later do the analysis correctly by himself.

ENTRY LEVEL BEHAVIOR: The learner must have completed the Basic Laboratory Skills course or have shown competence in the areas covered by this course. Course II, Basic Parameters, is not a prerequisite, but is desirable. He must have completed lessons 1 and 2 of this series. The learner must also be able to perform the following:

1. Adjust pH of a sample
2. Use a spectrophotometer
3. Plot a calibration curve

A INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will perform the Total Kjeldahl Nitrogen Determination by the micro method and Nesslerization, construct a calibration curve and do the final calculations according to the Effluent Monitoring Procedure.
2. Conditions: The learner will have at his disposal all the necessary equipment and reagents and the EMP.
3. Accepted Performance: The student must obtain an 80% on an Instructor checklist, or repeat until he does. (See C.3. Student Evaluation.) Note: The checklist includes a section for the completion of the student report form.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Total Kjeldahl Nitrogen Effluent Monitoring Procedure, and transparencies available from NTOTC, OT-9(N) Series of 9. Use:
   a. #6 Adding solution to form alkaline layer, and
   b. #7 % Transmittance and Absorbance Scale
2. Suggested Media:

C. INSTRUCTIONAL APPROACH: (Sequencing)

1. Presentation (15 minutes)

   All students steam-distilling the sample should be supervised very, very closely. The Instructor should demonstrate use of steam distillation apparatus prior to the student operation.

2. Student Performance: (3 hours)

   a. Student finishes digesting sample
   b. Student steam distills sample (micro method)
   c. Student prepares standards for calibration curve, (Nesslerization)
   d. Student, using standards and sample, plots calibration curve, and obtains ammonia concentration.
   e. Student calculates final Total Kjeldahl Nitrogen concentration
   f. NOTE: With a time frame of 3¼ hours, there is at least 30 minutes available at the end of the analysis for discussion of results.

3. Student Evaluation: Accepted performance as mentioned in A.3 above will be evaluated by the Instructor using checklist like the following:

EXAMPLE PERFORMANCE CHECKLIST
for the
TOTAL KJELDAHL NITROGEN DETERMINATION

A. Correct reagents used in digestion and distillation. YES___ NO___

B. Reagents delivered properly. YES___ NO___

C. Titration procedure performed correctly, (if Titration used.) YES___ NO___

D. Calibration curve prepared correctly, (if Nesslerization used.) YES___ NO___

E. Calculations performed correctly. YES___ NO___

F. Calculations evaluated by written quiz were correctly done. YES___ NO___

G. Final evaluation for record: SATISFACTORY______ UNSATISFACTORY_______

STUDENT________________________________

INSTRUCTOR______________________________

A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES

for the

NITROGEN, AMMONIA DETERMINATION

as applied in

WASTEWATER TREATMENT FACILITIES
and in the
MONITORING OF EFFLUENT WASTEWATERS

INSTRUCTIONAL PACKAGE WORKSHEET

National Training and Operational Technology Center
Municipal Operations and Training Division
Office of Water Program Operations
U. S. Environmental Protection Agency

CH.N.am.ipw.2c.6.77

PART II INSTRUCTIONAL PACKAGE WORKSHEETS

F. Nitrogen, Ammonia Determination

1. The Federal Register (FR) issuance in Outline 1 of this Guide lists one method for the isolation of Ammonia from a water sample, i.e., manual distillation at pH 9.5.

   > In a footnote, #4, it states: "Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies."

   The same FR issuance lists three manual methods for the measurement of the isolated Ammonia; nesslerization (for concentrations of 0.05-1mg/l), titration (for 1-25mg/l) and electrode (for concentrations from 0.05-1400 mg/l).

2. The distillation, nesslerization and titration methods for macro sample volumes have been written in the Effluent Monitoring Procedure (EMP) format entitled, "Nitrogen, Ammonia Determination." There is a separate EMP for the electrode method, "Determination of Ammonia by an Ammonia Selective Electrode." The distillation, nesslerization and titration methods for both macro and micro sample volumes are contained in another EMP, "Determination of Total Kjeldahl Nitrogen."

3. The reference source for the Nitrogen, Ammonia Determination EMP is 1974 EPA "Methods for Chemical Analysis of Water and Wastes," p. 159. Other FR sources for the procedure are:

   a. 14th ed., APHA, "Standard Methods for the Examination of Water and Wastewater," p. 410 (Distillation) and p. 412 (nesslerization);

   b. Part 31, 1975, "Annual Book of ASTM Standards," p. 237;

   c. Book 5, chap. A1, 1970, USGS, "Survey Techniques of Water-Resources Inventory," p. 116;

   d. 12th ed., AOAC, "Official Methods of Analyses of the Association of Official Analytical Chemists," p. 614.

4. The procedures in this EMP are for macro sample volumes with a choice of nesslerization or titration for the ammonia measurement.

5. In the following Instructional Package Worksheet the lesson plan is for distillation of a macro volume of a Wastewater Treatment Plant effluent sample followed by titration.

6. Students are to work in pairs to distill and titrate one sample. The IPW equipment and reagent requirements are based on this assignment. If you choose a different assignment, adjust quantitites accordingly.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrogen, Ammonia Determination

UNIT OF INSTRUCTION:

LESSON NUMBER: 1 of 1

ESTIMATED TIME: 1 1/2 hours

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The learner should know how to perform the distillation procedure for the determination of ammonia nitrogen since it is required by many National Pollutant Discharge Elimination System permits.

ENTRY LEVEL BEHAVIOR: The learner must be able to:

1. Perform basic mathematical computations.
2. Handle reagent chemicals safely.
3. Be familiar with laboratory apparatus.
4. Understand terms such as liter, milliliter, gram, and milligram.
5. Understand the term normality, to the extent that it is a chemical way of expressing concentration.
6. Perform weighings on analytical and trip balances.
7. Clean laboratory glassware.
8. Prepare chemical solutions.
9. Perform a titration.
10. Use a colorimeter.

A. INSTRUCTIONAL OBJECTIVE

1. Terminal Behavior: The learner will exhibit proper technique while determining the ammonia-nitrogen content of a wastewater treatment plant effluent.

2. Conditions: The learner will have the use of the attached EMP, and all chemicals and equipment listed in it.

3. Accepted Performance: The use of proper technique in performing the test will be judged by the instructor.

B. INSTRUCTIONAL RESOURCES

1. Available Media: 8 minute slide-tape, XT-67, Determination of Ammonia Nitrogen*

C. INSTRUCTIONAL APPROACH (Sequencing)

1. Classroom - discussion of the importance of the NH_3-N determination by the instructor and the use of an audio/visual unit, XT-67.

*Available from the U.S. Environmental Protection Agency, Office of Water Program Operations, National Training & Operational Technology Center, Cincinnati, Ohio 45268

2. Laboratory - determination of the NH_3-N concentration of a wastewater treatment plant effluent.

3. Classroom - discussion of calculations.

4. Classroom - final discussion of the procedure.

D. EQUIPMENT AND SUPPLY REQUIREMENTS:

(Macro distillation plus titration of 1 sample per 2 students)

1. For each student:

All equipment is shared; see below

2. Shared:

beaker, 50 ml (1 per 2 students)
beaker, 600 ml (1 per 2 students)
boiling beads, glass, 5 mm (6 per 2 students)
bottle, glass, 100 ml dispensing (1 per 2 students)
bottle, glass, 1000 ml with glass stopper (1 per 18 students)
bottle, glass, 1000 ml with rubber stopper (1 per 18 students)
buret, 50 ml with 0.1 ml graduations (1 per 2 students)
buret clamp (1 per 2 students)
cylinders, graduated, 100 ml (2 per 2 students)
cylinder, graduated, 500 ml (1 per 2 students)
distillation assembly (1 per 2 students). Diagram is in Kjeldahl Nitrogen EMP in equipment list.
  -Meker burner
  -800 ml Kjeldahl flask
  -Kjeldahl spray trap, connecting bulb, 55 mm
  -Graham or Allihn condenser, 16 inch
  -rubber stopper #3
  -rubber stopper #7
flask, Erlenmeyer, 500 ml (1 per 2 students)
flask, volumetric, 1000 ml with stopper (1 per 18 students)
stand, titration with procelain base for buret.

E. IPW REAGENT REQUIREMENTS:

(minimum amounts per student)

10 g suitable detergent
pH paper, strips, short range to test at pH 9.5
10 ml sodium hydroxide (1N)
25 ml borate buffer
50 ml boric acid solution (2%)
90 ml (maximum) standard sulfuric acid solution (0.02N)
3 drops mixed indicator
100 ml ammonia-free distilled water

A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES

FOR THE

DETERMINATION OF NITRATE-NITRITE NITROGEN AND OF NITRATE NITROGEN, CADMIUM REDUCTION METHOD

as applied in

WASTEWATER TREATMENT FACILITIES
and in the
MONITORING OF EFFLUENT WASTEWATERS

Instructional Package Worksheet

National Training and Operational Technology Center
Municipal Operations and Training Division
Office of Water Program Operations
U.S. Environmental Protection Agency

CH.N.n/n.ipw.2b.6.77

PART II INSTRUCTIONAL PACKAGE WORKSHEETS

G. Determination of Nitrate-Nitrite Nitrogen and of Nitrate Nitrogen, Cadmium Reduction Method

1. The Federal Register (FR) issuance in Outline No. 1 of this Guide lists two manual methods to determine Nitrate (as N): cadmium reduction and brucine sulfate (formation of a complex).

2. The cadmium reduction method has been written in the Effluent Monitoring Procedure (EMP) format entitled, "Determination of Nitrate-Nitrite Nitrogen and of Nitrate Nitrogen, Cadmium Reduction Method". The simpler equipment and techniques required are ideal if equipment and experienced personnel are limited, and the test is required intermittently. Furthermore, the method yields data for concentrations of nitrate plus nitrite, of nitrate and of nitrite.

   If only nitrate data is required, the time involved in using this method becomes a significant consideration when testing is done on a daily basis. The brucine sulfate method might be preferred. An EMP for the brucine sulfate method is in preparation.

3. The reference source for the Nitrate-Nitrite, Nitrate Nitrogen, Cadmium Reduction Method EMP is 1974 EPA "Methods for Chemical Analysis of Water and Wastes," p. 201. The only other FR source for the procedure is:

   a. 14th ed., APHA, "Standard Methods for the Examination of Water and Wastewater," p. 423.

4. The procedures in this EMP are for determining Nitrate plus Nitrite (asN) and Nitrite (as N). Nitrate (as N) concentrations are obtained by calculating the difference of the above. The EMP also includes the calculation of Nitrate (as NO_3) and of Nitrite (as NO_3) in case permits require these expressions of the data.

5. In the following Instructional Package Worksheet (IPW) the lesson plan is for determining nitrate plus nitrite, then nitrite in a synthetic sample, then doing the calculations listed above in 4.

6. Each student is to prepare one reduction column and then process one blank, one standard and one sample to determine nitrate plus nitrite (as N) concentration. He is also to process one blank, one standard and the same sample to determine nitrite (asN) concentration. The IPW equipment and reagent requirements are based on this assignment. If you choose a different assignment, adjust quantities accordingly.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrite + Nitrate Nitrogen, Nitrite Nitrogen, and Nitrate Nitrogen

UNIT OF INSTRUCTION: Summary of 11 Elements

ESTIMATED TIME: 400 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The testing of Nitrates and Nitrites (NO_3-N; NO_2-N; NO_3; NO_2) is required by many National Pollution Discharge Elimination System Permits.

ENTRY LEVEL BEHAVIOR: Learner must know how to weigh to 4 decimal places on an analytical balance, how to weigh to two decimal places using a triple beam balance, how to measure the volume of liquids with a pipet and a graduated cylinder, how to measure pH using a pH meter, how to make spectrophotometric measurements using a spectrophotometer, how to plot and interpret graphs, how to solve subtraction problems involving decimals and how to multiply and divide factors in a fraction.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will assemble the required equipment (including 1 reduction column), copperize cadmium and fill a reduction column with it, prepare a blank, one nitrate standard and a sample, pass each through the reduction column, add color reagent, measure the absorbances, construct a calibration curve using absorbances obtained by the class and obtain from it a mg NO_2 + NO_3-N/liter result for his reduced sample. He will also prepare a blank, one nitrite standard and a sample, add color reagent, measure the absorbances, construct a calibration curve using class absorbance values and obtain from it a mg NO_2-N/liter result for his non-reduced sample. He will calculate and report the efficiency of his column using the absorbance values for the two standards he tested. Then he will calculate for his sample mg NO_3-N/liter, mg NO_3/liter and mg NO_2/liter. He will give the instructor a completed data sheet and will have an opportunity to discuss the test, sample dilutions, applying column efficiency data and interferences to the test. All work must be done according to the EMP.

2. Conditions: being provided with classroom instruction, the required equipment and supplies, one sample, a copy of the EMP, a copy of the data sheet, two pieces of graph paper, and a total working time of 290 minutes.

3. Accepted Performance: The learner's techniques must be satisfactory according to an instructor's rating and the learner's answers must be the same as an instructor's answers calculated from the same data.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: EPA "Methods for Chemical Analysis of Water and Wastes," EMP on Determination of NO_3 + NO_2-N and NO_3-N, EMP (p. 48), Example Data Sheet, EMP (pp. 49 & 50), Calibration Graph forms for the test, Laboratory Equipment and Supplies, one 21 minute CCTV cassette on "Preparation of the Cadmium Reduction Column" and the following numbered overheads (11):

   a. #1 "Data obtained from the Cadmium Reduction Method"
   b. #2 EMP (p. 9) "Figure 1 Reduction Column"
   c. #3 Overview of steps for "Preparation of Reduction Column"
   d. #4 Overview of EMP Procedures E, F, G, H and L, "Determination of Nitrite Plus Nitrate Nitrogen"
   e. #5 Overview of EMP Procedures I, J, K and L, "Determination of Nitrite Nitrogen"
   f. #6 EMP (p. 48) "Example Data Sheet"
   g. #7 EMP (p. 49) Calibration Graph for Total NO_2 + NO_3-N with curve drawn from example absorbances on the EMP Data Sheet
   h. #8 EMP (p. 50) Calibration Graph for NO_2-N with curve drawn from example absorbances on the EMP Data Sheet
   i. #9 "Example of Calculating Column Efficiency"
   j. #10 EMP (p. 47) "Key to Data Sheet" which summarizes how to obtain five nitrogen values from the test data, using format of the EMP Example Data Sheet
   k. #11 A summary of "Removal of Interferences" from EMP Procedures D, F and J

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH: The method presented in the Effluent Monitoring Procedure (EMP) is according to the EPA Methods Manual. Procedures to determine total nitrite plus nitrate nitrogen by the cadmium reduction method and also nitrite nitrogen by colorimetry are included in entirety. The nitrate nitrogen in a sample can be calculated from the data obtained by these procedures. Since some permits require the data as nitrate and nitrite, the EMP includes calculating these concentrations from the data obtained using the EMP procedures.

1. Preparation for Instruction:

   a. Use list of equipment in D below to supply laboratory stations for each student.
   b. Make one glass reduction column for each student.
   c. Prepare all necessary reagents as listed in E below.
   d. Clean all glassware.
   e. Duplicate copies of the EMP data sheet, two for each student.
   f. Duplicate copies of the two EMP calibration graph forms for each.
   g. Check pH meters for proper operation.
   h. Check spectrophotometers for proper operation.

2. Sequencing:

   a. Lesson one - 70 minutes
   b. Break - 10 minutes

      (Alternative: A break of any length could come here since lesson one includes storage directions for the newly-prepared column.)

   c. Combination Presentation:

      Lesson two - 60 minutes
      Lesson three - 20 minutes
      Lesson four - 40 minutes

      There can be no break between lessons two and three since color development of the reduced blank and standard must begin within 15 minutes of the reduction process. To compensate for student differences in accomplishing lesson two, classroom presentations of lessons two and three can be combined into one session so students can do the laboratory work for each with no classroom break between.

      Since lesson four involves the same laboratory techniques as lessons two and three, all three lessons can be included in the same classroom session.

      (Alternative:

      (1) Lesson two - 60 minutes
      (2) No break
      (3) Lesson three - 20 minutes
      (4) Break - 15 minutes

         If lessons are presented in numerical order, this is the longest break you can allow from the end of lesson three to the end of lesson seven.

         If lesson seven [as applicable] immediately follows lesson four, a break up to 60 minutes may be allowed.

      (5) Lesson four - 40 minutes)

   d. Break - 15 minutes

      (NOTE: If the lessons are presented in numerical order and a 15 minute break was given after lesson three, there should be no break given now.)

      (Alternative: If lesson seven [as applicable] is to follow lesson four, this break can last up to 60 minutes. Subtract any break given after lesson three.)

   e. Successive Presentations:

      Lesson seven (as applicable) - 25 minutes
      Lesson eight (as applicable) - 25 minutes
      Lunch break - 60 minutes

This sequence was chosen so students can do the spectrophotometric measurements and calibration curve for the reduced nitrate standards and sample. This completes this half of the total workload and provides for a logically-placed long break.

(Alternative: If a long break is not desirable at this point, the lessons can be presented in numerical order. For this choice, only 15 minutes is available for a break from the end of lesson three to the end of lesson seven.)

f. Combination Presentation:

Lesson five - 35 minutes
Lesson six - 15 minutes

Lessons five and six involve the same laboratory techniques so the classroom presentations are easily combined.

(Alternative:

(1) Lesson five - 35 minutes
(2) Break - 15 minutes

If lessons are presented in numerical order, this break is allowable only if no break has occurred since the end of lesson three.

If lesson seven [as applicable] followed lesson four, a break up to 85 minutes may be allowed.

(3) Lesson six - 15 minutes)

g. Break - 10 minutes

If lessons are presented in numerical order, 15 minutes is the longest allowable break here provided no other break was given since the end of lesson three.

If lesson seven (as applicable) followed lesson four, a break up to 85 minutes may be allowed. Subtract any break time given after lesson five.

h. Lesson seven (as applicable) - 15 minutes

The classroom presentation and work time applicable to the reduced nitrate standards and sample was done earlier. Students only need a reminder in the classroom before doing the spectrophotometric measurements.

(Alternative: Lesson seven - 40 minutes. If lessons are presented in numerical order, all spectrophotometric measurements are done now.)

i. No break

(Alternative: If lessons have been presented in numerical order, only one 15 minute break can be given from the end of lesson three to the end of lesson seven. In this case, a break of any length could come now since the remaining lessons involve calculations and a summary session.)

j. Lesson eight (as applicable) - 15 minutes

See explanation under lesson seven.

(Alternative: Lesson eight - 40 minutes. If lessons were presented in numerical order, both calibration curves are constructed now.)

k. No break

(Alternative: A break of any length could be scheduled.)

l. Lesson nine - 25 minutes

m. Break - 10 minutes

(Alternative: A break of any length can be scheduled.)

n. Lesson ten - 25 minutes

o. No break

(Alternative: A break of any length can be scheduled.)

p. Lesson 11 - 30 minutes

D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

a.m. - 1 blank, 1 nitrate standard, 1 sample
p.m. - 1 blank, 1 nitrite standard, 1 sample

1. For Each Student:

1 apron
1 beaker, 150 ml
3 beakers, 250 ml
1 beaker, 400 ml
1 weighing boat (for cadmium)
1 wash bottle, squeeze type
1 glass column for reduction
1 cylinder, graduated, 50 ml
1 cylinder, graduated, 100 ml
3 flasks, 250 ml Erlenmeyer (If lessons are done in numerical order, you need 6.)
1 flask, 50 ml volumetric with stopper (if diluting sample)
2 flasks, 100 ml volumetric with stoppers (If lessons are done in numerical order, you need 4.)
1 pair rubber gloves
1 pair safety glasses

1 three inch length rubber hose (4 cm ID)
1 pH meter (avoid sharing; uses time)
1 pipet, Mohr, 10 ml
1 pipet, volumetric, 25 ml
1-3 pipets, volumetric, 50 ml
1 propipet bulb
2 clamps, one screw-type and one pinch cock
1 sieve, 60 mesh
1 spatula or stirring rod to transfer Cd to column
1 wad of glass wool for column
1 copy EMP data sheet
1 copy of the two EMP calibration graph forms

2. Shared:

1 balance (to weigh approximately 20 g Cd) per 3 students (You can pre-weigh.)
1 very large funnel containing fluted filter paper (for Cd rinsings) set in old bottle per 2 students
1 set volumetric pipets (1.0, 2.0, 5.0, 10.0 ml) per 4 students
1 one or two milliliter pipet graduated at 0.5 ml per 4 students
1 two milliliter volumetric pipet for each bottle of color reagent
1 spectrophotometer per 3 students
1 spectrophotometer cell per instrument
1 spatula by each balance (if students are to weigh out the Cd)
1 support stand per 2 students
1 snap-clamp buret holder per 2 students

E. IPW REAGENT REQUIREMENTS: (minimum amounts per student)

4 liters distilled water, nitrate-nitrite free
480 ml concentrated ammonium chloride - EDTA solution (300 ml for every 500 ml of dilute solution required)
800 ml dilute ammonium chloride - EDTA solution
12 ml color reagent (sulfanilamide, phosphoric acid, Marshalls reagent) Store in dark bottle in refrigerator.
~ 1 ml concentrated hydrochloric acid in dropper bottle for pH adjustment
~ 1 ml concentrated ammonium hydroxide in dropper bottle for pH adjustment (may not be required if sample is synthetic)
60 ml hydrochloric acid, 6 N
200 ml copper sulfate solution, 2%
11 ml potassium nitrate stock solution. Refrigerate. One liter is a convenient amount to prepare accurately.
10.0 ml potassium nitrite stock solution. Refrigerate. One liter is a convenient amount to prepare accurately.
~ 20 g cadmium, 40-60 mesh. If not available from a commercial source, file cadmium sticks with a coarse file and collect the granules which will pass a 10 mesh sieve and are retained on a 40, then a 60 mesh sieve. Use a hood. Wear rubber gloves and a mask.
about 100 ml each of pH buffers 4, 7 and 10 if students are to check the calibration of their pH meters

THE FOLLOWING SOLUTIONS ARE UNSTABLE. Prepare them just before use.

25 ml 1.00 mg NO_3-N/liter solution to activate the cadmium reduction column. Dilute 1 ml stock potassium nitrate solution to one liter.

15 ml potassium nitrate standard solution. One liter is a convenient amount to prepare accurately. (You might use up to 90 ml more to prepare 1 liter of sample. See below.)

15 ml potassium nitrite standard solution. One liter is a convenient amount to prepare accurately. (You might use up to 90 ml more to prepare 1 liter of sample. See below.)

50 ml sample of up to 1 mg NO_2 + NO_3-N/liter. You can use the standard solutions of each form to prepare this. Forty ml of each in one liter gives 0.4 mg/liter of each form for a sample of 0.8 mg NO_2 + NO_3-N/liter.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrite + Nitrate Nitrogen, Nitrite Nitrogen and Nitrate Nitrogen

UNIT OF INSTRUCTION: Preparation of Copper-Cadmium Reduction Column (EMP Procedure C)

LESSON NUMBER: 1 of 11

ESTIMATED TIME: 70 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: To determine NO_3-N in a sample, the learner must prepare a reduction column to reduce nitrate to nitrite.

ENTRY LEVEL BEHAVIOR: Learner must be able to measure the volume of liquids with a graduated cylinder.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will set up a functioning copper-cadmium reduction column according to the steps in EMP Procedure C as stated or described in the information column.

2. Conditions: Learner will be given the EMP, the necessary equipment, two copies of data sheet, 40 minutes and no assistance.

3. Accepted Performance: Learner must accomplish terminal behavior to the satisfaction of an instructor rating. (Particularly note the proper flow rate of 7-10 ml/minute.)

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of EMP; three overheads: (1) "Data Obtained from the Cadmium Reduction Method," (2) EMP "Figure 1 Reduction Column," p. 9, (3) overview of steps for "Preparation of Reduction Column;" 21 minute CCTV cassette on "Preparation of the Reduction Column"

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: 30 minutes

   a. Lecture - Introduction

      - Use Training Guide I to introduce the test and the reasons why nitrogen forms are important to water quality.
      - Use overhead (1) "Data Obtained..." to establish the two determinations students will do in the laboratory.
      - Show overhead (2) of EMP Figure of the Reduction Column.
      - Use overhead (3) to give overview of steps to prepare the column. Stress importance of flow rate.
      - Caution re using cadmium (use gloves, do not inhale).

Preceding page blank

b. Assignment: Read from above

A. Instructional Objective, 1. Terminal Behavior

c. Show CCTV cassette on preparing the column.

d. Question period on material presented

2. Student Performance and Evaluation: (40 minutes)

As stated in Instructional Objective above, by instructor rating.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrite + Nitrate Nitrogen, Nitrite Nitrogen and Nitrate Nitrogen

UNIT OF INSTRUCTION: Preparation of Nitrate Working Standards and Reduction of Nitrate to Nitrite (EMP Procedures E and F)

LESSON NUMBER: 2 of 11

ESTIMATED TIME: 60 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: This determination requires a calibration curve derived from nitrate working standard solutions which have been reduced to the nitrite form.

ENTRY LEVEL BEHAVIOR: Learner must know how to use a pipet, a volumetric flask, and a single-electrode pH meter.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will prepare a blank and one assigned nitrate working standard solution and pass each solution through the reduction column to reduce nitrate-nitrogen to nitrite-nitrogen according to the steps in EMP Procedures E and F as stated or described in the information column.

2. Conditions: Learner will be given the equipment, the EMP, 50 minutes, and no assistance.

3. Accepted Performance: Learner will perform procedures E and F to the satisfaction of an instructor rating. (Particularly note pipetting techniques and dilution to volume.)

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of EMP; two overheads: (1) "Data Obtained from the Cadmium Reduction Method" and (4) Overview of steps for "Determination of Nitrite Plus Nitrate Nitrogen" (E, F, G, H, L)

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: Briefing (10 minutes)

   a. Use overhead (1) to stress that this is the beginning of the Determination of Nitrite Plus Nitrate Nitrogen.

   b. Use overhead (4) to give overview of EMP ProceduresE, F, G, H and L used to do this determination.

   c. Assignment: Read from above

      A. Instructional Objective, 1. Terminal Behavior

   d. Assignment: Have students read Procedures E and F.